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THE JOURNAL

OF THE

Society of Dyers and Colourists

Volume 66—Number 4

APRIL 1950

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Proceedings of the Society

Report of the Committee on the Dyeing Properties of Wool Dyes

Introduction

Following the publication of the Report of the Committee on the Dyeing Properties of Direct Cotton Dyes¹, Dr. C. M. Whittaker, then President of the Society, suggested that the Committee should extend their work to fibres other than cotton. After due consideration the Direct Cotton Dyes Committee reported that they concurred in the suggestion that similar work could usefully be carried out on wool dyes but that they considered that it would be preferable to appoint a separate committee for this purpose.

Council considered this recommendation on 20th November 1946, and proceeded to set up a Committee, comprising representatives of dye users, dye manufacturers, and research workers, to consider the problem relating to wool dyes and to proceed with such work as was thought fit. (The constitution of the Committee is given in Appendix V.)

The new Committee held preliminary discussions on 4th February and 24th March 1947, when the dye-user members of the Committee drew attention to the inadequacy of the information given in dyemakers' pattern cards and to the variation in the dyeing methods employed by the various dyemakers. They expressed the view that the Committee could do useful work if only in standardising these methods. It was finally agreed that the Committee should try in the first instance to draw up simple methods for determining the dyeing and migrating properties of wool dyes.

The work has, in fact, been largely concerned with this aspect, namely the standardisation of existing methods for determining the dyeing and migrating properties of dyes on wool. To facilitate the use of the prescribed tests they have specified appropriate standards of control, and have formulated a grey scale (S.D.C. Grey Scale No. 2) with which to assess the results of the migration test.

During the course of the work the Committee have met numerous difficulties arising largely from a lack of reproducibility between the various laboratories concerned. Difficulties of this nature have prolonged the work, and the Committee have therefore felt it desirable to conclude their task at this stage rather than to proceed with their original

programme, which included the examination of the dyeing properties of premetallised and chrome dyes.

The recommendations which are made by the Committee on the testing of acid dyes are given in Part I of this report. The work on which these are based is described in some detail in Part II, partly to give an adequate background to the recommendations and partly because a number of problems of general interest were investigated during the course of the Committee's work.

The Committee felt that it would increase the practical value of the report if they were to give the results obtained with a range of acid dyes when subjected to the proposed tests. They have been able to do this through the co-operation of one of their members, in whose laboratory the tests leading to the results given in Appendix VI have been carried out.

The Committee held eighteen meetings between February 1947 and January 1949, with a further meeting in November 1949.

Part I—Recommended Methods of Test

The tests which the Committee recommend fall into two parts. The first part comprises a group of five tests which are intended to determine the optimum conditions under which a given acid dye can be applied to wool so as to give an economic degree of exhaustion. The second part consists of one test which is designed to give a measure of the ability of a dye to migrate in the dyebath from one part of the dyed material to another.

A—DYEING METHOD

Dyeings are made on wool serge, which has been scoured and prepared for dyeing in a suitable manner*. The amount of dye used is sufficient to yield a dyeing of medium depth (see Appendix I). Other additions to the dye liquor, which are based on the weight of the wool, are given below. Details of the solutions to be used in making up the dye liquors are given in Appendix II, and suitable apparatus in which to carry out the tests is described in Appendix III.

* Wool serge for carrying out these tests may be purchased from the Society of Dyers and Colourists, 32-34 Piccadilly, Bradford, Yorks.

Method

I	...	8.8%	Sodium sulphate (anhydrous)* and 3% sulphuric acid (168° Tw.)
II	...	8.8%	Sodium sulphate (anhydrous) and 2% formic acid (85%)
III	...	8.8%	Sodium sulphate (anhydrous) and 2% glacial acetic acid
IV	...	5%	Ammonium sulphate
V	...	50%	Sodium sulphate (anhydrous)

Distilled or condensed water is used, and the liquor : wool ratio is maintained throughout at 100 : 1 by any convenient means, provided that free escape of ammonia is permitted in Method IV.

The fabric is wetted out in hot distilled water only, and squeezed. It is entered into the dye liquor, which is heated from 40°C. to the boil in 30 min. and then boiled at a moderate rate for 1 hr., after which the fabric is removed. The dye liquor is filtered whilst still hot in order to remove small fibres, and cooled. The pH of the dye liquor is measured, and the degree of exhaustion of the dye is then determined by one of the methods given below.

The pH of the dye liquor should lie within the following limits—

Method		pH
I	...	2.8-3.1
II	...	3.0-4.3
III	...	4.7-5.1

If it lies outside these limits the dyeing should be repeated.

In order to select the dyeing method most suitable for use with the dye under test a degree of exhaustion of 90% has been chosen arbitrarily as the criterion (in practice a tolerance of $\pm 2.5\%$ is allowed for experimental variation). If 90% exhaustion is reached with two or more of the methods, the one employing the least acidic conditions (i.e. the one having the higher number) is chosen. This applies only to Methods I-IV; Method V is designed to assess the suitability of a dye for single-bath union dyeing.

NOTE—Certain dyes, mostly of the triphenylmethane class, may give less than 90% exhaustion even when Method I is used; they will, of course, be classified as Method I dyes.

Estimation of Exhaustion

This is carried out by estimating the dye in the residual dye liquor either (a) colorimetrically or (b) by means of exhaust dyeings.

(a) COLORIMETRIC METHODS—Both photoelectric and visual methods may be employed. With both these methods the solution to be measured should contain at least 25% pyridine, and with photoelectric methods a light filter having a maximum transmission in the region of the maximum optical density of the solution should be used. When visual methods are employed, the standard solution should be brought to a similar condition as regards pH and salt concentration as the residual dye liquor by means of appropriate additions of acid and sodium sulphate.

(b) EXHAUST DYEINGS—These are carried out by entering a piece of undyed serge of the same weight as the original piece into the residual dye liquor and dyeing in the usual manner, care being

taken that complete exhaustion and good penetration are obtained. In order to assess the amount of dye on the exhaust dyeing, the latter is compared with two supplementary samples dyed with 7.5% and 12.5% of the depth of the parent dyeing, these corresponding to an exhaustion in the parent dyeing of 90.0 + 2.5% and 90.0 - 2.5% respectively. In order to fall within these limits of exhaustion the depth of the exhaust dyeing must therefore be equal to or between those of the supplementary dyeings.

Control Dyeing

In order to facilitate the standardisation of the dyeing conditions, a dyeing of 1.4% Kiton Fast Red R (for equivalent dyes see Appendix VI) is carried out by Method I. At the completion of the dyeing the degree of exhaustion, determined preferably by colorimetric estimation of the residual dye in the cooled dye liquor, should be not less than 87.5% nor more than 92.5%. The final pH of the residual dye liquor should be 2.8-3.1.

B—MIGRATION TEST

A sample of undyed wool serge ("Sample 1"), which has been scoured and prepared for dyeing in a suitable manner, is dyed to a medium depth (see Appendix I) with the additions prescribed in one of the Dyeing Methods I-V (see Section A above). The dyeing method chosen should be that appropriate to the dye under test, as already determined by the methods of Section A.

The fabric is wetted out in hot distilled water only, and squeezed. It is entered into the dye liquor, which is heated from 40°C. to the boil in 30 min. and then boiled at a moderate rate for 1 hr. Distilled or condensed water is used, and the liquor : wool ratio is maintained throughout at 100 : 1 by any convenient means, provided that free escape of ammonia is permitted in Method IV (see Appendix III).

A second sample of undyed wool serge ("Sample 2"), of the same weight as Sample 1, is similarly and concurrently treated, except that the dye is omitted. At the completion of the 1-hr. boiling period, half of Sample 2 is removed from the liquor and discarded, and is replaced by half of Sample 1, taken directly from the dyebath. The liquor is boiled at a moderate rate for a further hour, the liquor : wool ratio being maintained at 100 : 1.

After rinsing and drying, Samples 1 and 2 are assessed by comparing their contrast with those on the Grey Scale No. 2 (see Appendix IV). By this means a numerical value is given to the degree of migration.

NOTE—When a sample is available which has been dyed with the dye under test in a suitable depth of dyeing, the following shortened method may be employed and will lead to a similar result—

Equal weights of dyed and undyed fabric are wetted out in hot distilled water only, squeezed, entered into a boiling liquor, to which additions (based on the total weight of wool) appropriate to one of the Dyeing Methods I-V have been made, and boiled for 1 hr., the liquor : wool ratio being maintained at 100 : 1 by any convenient means, provided that free escape of ammonia is permitted

* 8.8% Sodium sulphate (anhydrous) = 20% Glauber's salt.

in Method IV. After rinsing and drying the samples are assessed as above.

Control

In order to control the method of test, in particular the rate of boiling subsequent to the interchange of the samples, the migration test is carried out using a dyeing of 0.85% Brilliant Scarlet R (for equivalent dyes see Appendix VI) which has been dyed by Method III.

The samples resulting from the subsequent migration test should indicate a migration of 3 when assessed by means of the Grey Scale No. 2.

Part II—An Account of the Work of the Committee

The Committee first addressed a questionnaire to dyemakers asking for information on three points, viz.—

- (1) The tests used to determine the optimum dyeing method for a particular dye
- (2) The significance of the rating for "level dyeing" listed in pattern cards
- (3) The assessment of the "neutral-dyeing property" of a dye.

Information on these items was received from seven dyemakers. A fair measure of agreement was shown, the practice of each dyemaker being to carry out four or five dyeings under different conditions ranging from neutral to strongly acidic. Six dyemakers also gave details of their "level dyeing" tests, which in all cases measured the extent of migration of the dye from a fully dyed to an undyed pattern. Neutral-dyeing property was stated to be assessed in terms of the colour yield of a dyeing made with an addition of Glauber's salt.

Several members of the Committee expressed the view that the optimum dyeing method for a particular dye could be most readily ascertained by making two dyeings at different pH values, e.g. 4.7 and 5.9, so that dyes could be broadly classified into three groups, viz.—

- (I) Those requiring sulphuric acid for exhaustion
- (II) Those exhausting in the presence of acetic acid
- (III) Neutral-dyeing dyes.

Dyes classed under (I) would not exhaust adequately in either dyeing, dyes classed under (II) would exhaust in one dyeing only, and dyes classed under (III) would exhaust in both dyeings. This method, however, did not meet with general support.

The Committee, therefore, drew up five provisional dyeing methods, based on accepted practice, as follows—

Dyeings are made on wool scoured and prepared for dyeing in the usual manner. The wool is entered into the dye liquor at 40°C., and the dye liquor brought to the boil in half-an-hour and maintained at the boil for a further hour. The dyeing is of medium depth (see Appendix I). Distilled or condensed water is used throughout, and the liquor : wool ratio is maintained at 40 : 1.

Free escape of ammonia is to be permitted in Method IV.

Method

I	...	20% Glauber's salt + 3% sulphuric acid (168° Tw.)
II	...	20% Glauber's salt + 2% formic acid (85%)
III	...	20% Glauber's salt + 2% glacial acetic acid
IV	...	5% Ammonium sulphate
V	...	20% Glauber's salt (primarily for union dyes)

ADDITIONS TO DYE LIQUOR

It was agreed that the criterion of the suitability of any method was that it should give an exhaustion of at least 90% under the least acidic conditions.

NOTE—It will be seen that in the methods as given above a liquor : wool ratio of 40 : 1 has been used, whereas in the prescribed tests given in Part I a ratio of 100 : 1 is given. This change was made at a later stage in the Committee's work during investigations relating to the migration test, when it was found advantageous to change to the higher ratio as explained in a later section (see p. 217). The Committee felt that similar advantages would also apply to the dyeing methods, which were therefore amended.

The Committee realise that a liquor : wool ratio of 100 : 1 is higher than is normally used in practice. They consider, however, that this fact is not relevant to a system of tests which is designed to classify wool dyes. Moreover, they are satisfied that the tests as now proposed give results which correspond to practical experience in the various types of wool dyeing.

METHOD OF DETERMINING THE DEGREE OF EXHAUSTION OF THE DYE LIQUOR

Two methods were suggested for estimating the exhaustion, viz.—

(i) A sample of wool equal in weight to that used in the original dyeing is dyed to exhaustion in the liquor remaining from each of the above dyeings. These exhaust dyeings are then compared with a further dyeing made with one-tenth the amount of dye used in the original dyeing.

(ii) One-tenth of the weight of wool used in the original dyeing is dyed to exhaustion in the remaining dye liquor and compared with the original dyeing.

When these tests were carried out it was found that it was difficult to obtain satisfactory exhaustion using a sample one-tenth the weight of the original, owing to the high liquor : wool ratio (400 : 1). As the visual assessment was found to be easier using a sample of equal weight, this method was adopted.

INFLUENCE OF RESIDUAL ALKALI IN WOOL

The influence on the dye exhaustion of the alkali remaining in wool after scouring was investigated. Dyeings were carried out using wool which had been treated in 0.2% sodium carbonate solution at 40°C. for 20 min. and not rinsed, the pH of the water extract of the wool after this treatment being 11.0–11.5. Dyeings made on this wool in the presence of 2% or less acetic acid gave slightly less exhaustion than those made on normally scoured wool. Dyeings made on various samples of normally scoured wool gave no significant differences, and it was concluded that the effect of residual alkalinity could be neglected if the pH of the water extract of the wool was less than 10.

In case of doubt, however, it was suggested that treatment in a boiling ammonium acetate solution containing 1% on the weight of the wool for 10 min. would eliminate all effects due to residual alkalinity, this treatment having been shown to be effective in eliminating dyeing differences when using the excessively alkaline wool described above.

LOSS OF FORMIC AND ACETIC ACIDS DURING DYEING

Experiments were carried out to determine the loss of volatile acids during dyeing operations. Dyeings were made using 1% dye, 20% Glauber's salt, and 2% acid (glacial acetic acid or 85% formic acid) in a 50 : 1 liquor ratio. The wool, after wetting out, was entered into the dye liquor, which was raised to the boil in 20 min. and boiled for 1 hr. The dyeings were made both under reflux and in open beakers. Table I gives the amount of acid, expressed as a percentage of that originally present, which was lost by distillation, and also the final pH of the dye liquor.

	Open Beaker Loss of Acid (%)	Final pH of Dye Liquor	Reflux Loss of Acid (%)	Final pH of Dye Liquor
Acetic acid ...	15.0 17.7	4.65	0.0	4.4
Formic acid ...	9.4 10.3	4.1	4.4	3.9

Measurements of the degree of exhaustion of four dyes were then made with acetic and formic acids, under reflux conditions and in open beakers. The results obtained are given in Table II.

	Exhaustion (%)			
	Acetic Acid		Formic Acid	
	Open Beaker	Reflux	Open Beaker	Reflux
Solway Blue RNS (ICI) ...	71.4	74.3	89.7	89.7
Coomassie Blue RLS (ICI)...	99.0	99.2	100.0	100.0
Carbolan Blue BS (ICI) ...	99.8	99.7	99.8	99.7
Disulphine Blue ANS (ICI)...	83.7	82.1	88.6	87.5

Thus, despite the much greater loss of these acids when dyeing in an open beaker, there was no significant difference in the degree of exhaustion of the above four dyes, either under reflux or in open beakers. It was concluded, therefore, that with the dyeing times already chosen no further account need be taken of the volatility of these acids.

EFFECT OF ADDING GLAUBER'S SALT IN DYEING METHOD IV

The rates of dyeing of three dyes were compared when dyed on wool serge in the presence of (i) 5% ammonium sulphate only, and (ii) 5% ammonium sulphate and 20% Glauber's salt, the liquor ratio being 40 : 1 throughout. The dyeings were begun at 40°C., brought to the boil in 30 min., and boiled for a further 5 min. Samples of the dye liquor were taken at 5-min. intervals for the colorimetric estimation of the amount of dye remaining in the dye liquor. The results are given in Table III.

These results showed that the addition of Glauber's salt had had no significant effect on the ultimate exhaustion of any of the three dyes, although differences were observed in the rate of absorption at temperatures below the boiling-point.

Time (min.)	Tem- perature	Exhaustion (%)			
		0.9% Wool Glauber's salt—	Brilliant Blue BL (Gy)	1% Carbolan Blue BS (ICI)	1.1% Polar Brilliant Blue GAW (Gy)
		With- out	With	With- out	With
5	50°C.	21.9	27.5	17.5	11.5
10	60°C.	35.3	40.9	25.0	18.3
15	70°C.	48.5	57.3	33.8	27.2
20	80°C.	58.6	66.0	47.6	40.9
25	90°C.	76.9	81.1	75.1	69.2
30	Boil	86.4	85.4	88.1	82.1
40	Boil	92.0	89.4	96.4	95.0

It was agreed that this dyeing should be carried out in the presence of ammonium sulphate only, as being the more usual method for dyes to which it would be applicable.

DYEING METHOD V

Some experiments were carried out in order to determine whether or not salt additions should be made in this dyeing method. Tests were carried out with three dyes, two being neutral-dyeing acid dyes and the third an acid dye with moderate neutral-dyeing properties. The dyeings were made by the standard method employing a 100 : 1 liquor : wool ratio, the salt addition being both sodium sulphate and sodium chloride. The degree of exhaustion of the dye was estimated by matching against fully exhausted dyeings of known strength. The results are given in Tables IV and V.

Effect of Sodium Sulphate on Exhaustion (%)				
Sodium sulphate (anhydrous), %	0	20	40	100
Sulphonol Red PG (YDC) ...	75	80	85	90
Coomassie Brown GS (ICI) ...	75	80	90	95
Disulphine Green BS (ICI) ...	25	30	30	30

Effect of Sodium Chloride on Exhaustion (%)				
Sodium chloride, %	0	20	40	100
Sulphonol Red PG (YDC) ...	75	80	85	90
Coomassie Brown GS (ICI) ...	75	80	90	95
Disulphine Green BS (ICI) ...	25	30	32.5	35

The results show that the neutral-dyeing acid dyes are sensitive to additions of salts, increased exhaustion being promoted by increasing the salt concentration. The effects of sodium sulphate and sodium chloride were similar, good exhaustion requiring at least 40% of salt. Further tests were carried out to determine the effect of similar salt additions on the migrating properties of dyes of this type. These tests were carried out with a much wider range of dyes of the neutral-dyeing type, and the results showed that, in the main, salt addition had very little effect on their migrating properties.

On the basis of this evidence, it was agreed that 50% anhydrous sodium sulphate should be used in Method V.

MIGRATION TEST

It was agreed that a standard method should be devised for assessing the degree of migration or the "levelling" properties of a dye.

It should be stated at the outset that there were two lines of thought on the conditions to be employed. One member of the Committee suggested that the test should be carried out in the presence of sulphuric acid whatever the type of dye under test, maintaining that such a test

would separate those dyes which did level, and which were therefore suitable for piece dyeing, from those which did not level. The other view, which received more support, was that there did not appear to be any point in carrying out a migration test on a particular dye under conditions which would never be used in practice, and that the migration test should therefore be carried out under the optimum dyeing conditions for the particular dye under test.

A provisional method for carrying out the test was then drawn up. The test consisted in dyeing a sample of wool serge with the dye under test by the appropriate dyeing method. At the same time another sample of serge of equal weight was treated under the same conditions but in the absence of any dye. Half of the undyed serge was then removed and replaced by half of the dyed serge taken directly from the dyebath. The two samples were then boiled together at a moderate rate for an hour, the liquor : wool ratio being maintained at 40 : 1 throughout. Using this method, members carried out tests with a range of acid dyes. In general the differences between the results of migration tests carried out in the presence of sulphuric acid and those carried out under the optimum dyeing conditions were small.

Other tests which were carried out indicated—

- (i) that the migration test was not influenced by drying the dyed wool prior to the test
- (ii) that pretreating the undyed wool under the same conditions as the dyed wool was not essential
- (iii) that the results obtained under reflux when using the apparatus illustrated in Appendix III were similar to those obtained in open beakers.

It was also pointed out that the full test as given in Part I was unnecessarily time-consuming, and that the same results would be obtained by using a much shorter procedure when a suitable dyeing of the dye under test was already available. It was decided, however, that the standard method should be the complete one, but that it should be pointed out that similar results could be obtained by using a shortened procedure.

It was apparent at this stage that the degree of reproducibility between the results obtained by members when using the same dye was unsatisfactory, the difference in the assessments amounting in some cases to as much as two grades on the 1-5 scale. Some experimental support was found for the view that variations in the wool used were responsible, but one member who carried out tests on the materials used by six other members was unable to repeat their results.

A more comprehensive experiment, in which five members carried out migration tests with two dyes using eight different samples of white material, indicated that the differences in the results of the migration tests could, to a large extent, be accounted for by differences in the material used for the tests. Least migration was obtained when worsted yarn was used and most with woollen flannel, worsted serge giving an intermediate effect. The degree of migration obtained with the

different samples was shown to increase with the degree of damage as indicated by staining the samples with a cold solution of Kiton Red G². The pH of water extracts of the samples also showed variation, but it was not thought that these would give rise to differences in the dyeing or migration tests, bearing in mind the work which had been carried out on the effect of alkali on dye absorption.

These results led directly to the suggestion that a standard material should be available for dyeing and for migration tests. It was agreed that for experimental work fabric was generally more suitable than yarn, and it was recommended that a stock of a suitable light-weight serge should be obtained and supplied by the Society on request.

Further migration tests were carried out using the same material, but the results were again variable. It was shown that stirring and rate of boiling had a considerable effect on the degree of migration.

A more detailed investigation of the conditions of the migration test, particularly of the influence of time and liquor ratio, was then carried out by three members. As the result of this work it was agreed that the test should be carried out for 1 hr. at 100 : 1 liquor ratio instead of for 30 min. at 40 : 1 liquor ratio as originally suggested. These conditions facilitated level dyeing and penetration, especially when the tests were carried out without manual stirring, and this enabled a more satisfactory assessment to be made.

It was also shown that the rate of boiling had a pronounced effect on the result of the test. It was thought that this might be controlled by a method which had been examined by the Committee on the Dyeing Affinity of Regenerated Cellulose, namely by counting the drops which fall from the finger condensers of the reflux apparatus used for carrying out dyeing tests (Fig. 1). After a thorough examination of this, however, it was abandoned in favour of a simple instruction that the test should be carried out in such a way as to give a specific result with a particular dye (see section on Standardisation).

Other suggestions made during the course of the work on the test were—

(i) That a very vigorous boil should be employed, as this would be more reproducible in its effect from one laboratory to another. The objection to this was that it would reduce the difference in the levelling properties within the more readily levelling group of dyes.

(ii) That the test should be carried out at 90°C. with continuous stirring. This method gave unsatisfactory results in that the originally undyed sample was uneven and badly penetrated, and therefore difficult to assess.

ASSESSMENT OF THE MIGRATION TEST

It was obvious in the early stages of the experimental work on the migration test that it would be necessary to assess the degree of migration by a visual method.

It was suggested that a grey scale could be used as a reference standard of contrast for this purpose. A scale of this type had already been devised and

adopted by the Society's Fastness Tests Committee³ for assessing the degree of staining on white samples (S.D.C. Grey Scale No. 1). After trials with different ranges, it was finally decided to employ a scale of five grades, in which grade 5 represented complete levelling, and grade 1 the lowest degree of migration which was considered to be of commercial importance. This scale was tried by several members of the Committee and, being found to give satisfactory results, it was adopted as the reference standard for the migration test. It was decided to ask the Society to prepare and distribute the scales to ensure uniformity. Details of the preparation of this scale (S.D.C. Grey Scale No. 2) are given in Appendix IV.

STANDARDISATION

In the course of the experimental work on the dyeing methods and the migration test, it became increasingly obvious that it would be necessary to prescribe one or more dyes which could be used for standardising those factors of the tests which experience had shown to be the most variable when carried out by different workers, e.g. rate of boiling. In this way individual workers would have the means of determining whether or not they were carrying out the tests in the specified manner.

Standard for Dyeing Method

After tests with a number of dyes, Kiton Fast Red R was selected as the most suitable dye. A group of four members of the Committee made 1.4% dyeings with this dye on the same white wool serge, both in open beakers and under reflux, under the conditions of Method I. The residual dye liquor from each dyeing was divided into four portions and distributed to each of the other members of the group, the residual dye content and pH of each sample of dye liquor being then estimated by each member. The results of comprehensive tests are given in Table VI, in which the four members are referred to as A, B, C, D.

TABLE VI
Exhaustion (%) of 1.4% Kiton Fast Red R
(Method I—100:1 liquor ratio)

Dyeings by Measurement by	Method	A		B		C		D	
		Dye (%)	pH	Dye (%)	pH	Dye (%)	pH	Dye (%)	pH
A	Open beaker	90.0	2.95	89.0	2.98	88.5	2.96	90.2	2.95
	Reflux	88.2	2.93	89.2	2.94	90.1	2.94	91.3	2.95
B	Open beaker	89.5	2.95	89.1	2.96	88.5	2.95	90.5	2.96
	Reflux	88.7	2.93	89.3	2.96	90.1	2.94	91.7	2.96
C	Open beaker	90.4	2.89	89.4	2.89	88.7	2.90	90.6	2.90
	Reflux	89.6	2.89	89.6	2.83	90.1	2.89	91.6	2.90
D	Open beaker	91.0	2.90	89.0	2.90	89.0	2.94	91.0	2.90
	Reflux	90.0	2.89	90.0	2.86	91.0	2.93	91.0	2.91
Mean—	Open beaker	90.2	2.92	89.3	2.96	88.7	2.94	90.6	2.93
	Reflux	89.2	2.91	89.5	2.90	90.3	2.93	91.4	2.93
Mean values	Open beaker	Dye absorbed 90.0%; pH 2.94							
	Reflux	Dye absorbed 89.7%; pH 2.91							

These results gave a clear indication of the reproducibility between one laboratory and another. It was agreed that the exhaustion of a 1.4% dyeing of Kiton Fast Red R (CAC) when dyed under the conditions of Method I should be $90.0 \pm 2.5\%$ and that the pH of the residual dye liquor in this method should be 2.8–3.0 (this was subsequently altered to 2.8–3.1).

Standard for Migration Test

On examining selected dyes, the most satisfactory results for this purpose were given by a 0.85% dyeing of Brilliant Scarlet R dyed by Method III. It was agreed that the conditions of the test should be such that the migration of this dye was 3, when assessed on the grey scale.

"STRIKE" TEST

During the work on the migration test it was noted that the difference in degree of migration given by certain dyes was less than might be anticipated in view of the known differences in their dyeing behaviour. Thus it was found that the results of the tests of Brilliant Scarlet R and Xylene Light Yellow 2G were not greatly different, although the latter was known to be a dye with better level-dyeing properties. The view was expressed that migration was not the only factor determining the level-dyeing properties of a dye, and that attention should also be paid to the way in which the dye was taken up by the wool in the early stages of dyeing, i.e. the "strike". It was pointed out, however, that these two dyes would be distinguished from one another by the dyeing tests. Nevertheless, a rate-of-dyeing test was examined. The absorption of five dyes was measured at intervals whilst the temperature was raised to boiling-point. The test, which involves accurate control of temperature increase, was not easy to carry out, and the results were inconsistent.

One further attempt was made to get information on the "strike", by employing a modification of the migration test. This involved entering the second sample of wool at an earlier stage than was prescribed in the accepted migration test and completing the test in the original dye liquor, as contrasted with the liquor to which no dye had been added. Tests were carried out by entering the second sample (a) as soon as the liquor had reached the boil, (b) after 15 min. at the boil, and (c) after 30 min. at the boil. Thus the second pattern absorbed some of the dye remaining in the dyebath at the time of its entry in addition to the dye which migrated in the later stages.

After a number of tests had been carried out, it was agreed that it was not possible to derive useful information in this manner, since this involved attempting to measure two properties simultaneously.

After carefully considering the position reached at this stage, the Committee decided that the development of a simple test which would give information on the rate of dyeing would require much further work, which they did not feel justified in undertaking. It was therefore decided not to proceed further, but to issue a Report covering the tests already drawn up.

APPENDIX I

Standard Dyeings to indicate Suitable Depths for Testing Dyes

The following dyeings represent suitable depths for carrying out the tests described in the Report. The amount of dye under test should be chosen so

as to give a depth of dyeing approximately equal to these controls—

0.8% Kiton Fast Yellow 2G (CAC)
1.0% Kiton Fast Orange G (CAC)
2.1% Kiton Red G (CAC)
2.7% Alizarine Fast Rubine R (CAC)
1.2% Kiton Fast Brown 2RL (CAC)
1.0% Fast Acid Violet B (CAC)
1.0% Brilliant Acid Blue G (CAC)
1.5% Benzyl Green B (CAC)
1.7% Alizarine Sapphire Blue 4G (CAC)
2.0% Alizarine Fast Grey G (CAC)
6.0% Fast Acid Navy Blue LBD (CAC)
14.0% Acid Black PR (CAC)

APPENDIX II

Preparation of Solutions used in Tests

It is recommended that the following solutions be used in preparing the dye liquors—

ACETIC ACID—Dilute 4 g. glacial acetic acid to 1 litre with distilled water. 5 ml. of this solution per gram of wool gives 2% glacial acetic acid, as required in Method III.

AMMONIUM SULPHATE—Dissolve 10 g. ammonium sulphate in distilled water and make up to 1 litre. 5 ml. of this solution per gram of wool gives 5% ammonium sulphate, as required in Method IV.

FORMIC ACID—Dilute 4 g. formic acid (85%) to 1 litre with distilled water. 5 ml. of this solution per gram of wool gives 2% formic acid (85%), as required in Method II.

SODIUM SULPHATE—Dissolve 17.6 g. anhydrous sodium sulphate or 40 g. Glauber's salt in distilled water and make up to 1 litre. 5 ml. of this solution per gram of wool gives 8.8% sodium sulphate (anhydrous), as required in Methods I, II, and III. For Method V, 28.5 ml. per gram of wool gives 50% sodium sulphate (anhydrous).

SULPHURIC ACID—Add 6 g. sulphuric acid (168°Tw.) to distilled water and make up to 1 litre. 5 ml. of this solution per gram of wool gives 3% sulphuric acid (168°Tw.), as required in Method I.

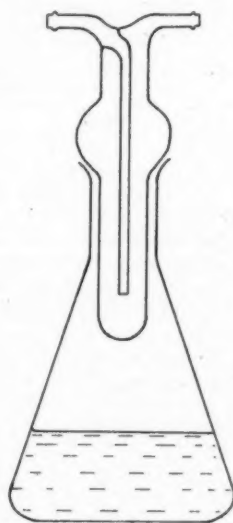


Fig. 1

1 in.

APPENDIX III

Apparatus

It is recommended that dyeings by Methods I, II, III, and V should be carried out in the apparatus devised by the Direct Cotton Dyes Committee¹. This consists of a conical flask of resistance glass, in the mouth of which is inserted a water-cooled condenser*, as shown in Fig. 1. The flask may be heated by any convenient method, such as an oil-bath or a hot plate. It is convenient to use a 600-ml. flask with a 4-g. sample of serge, or a 500-ml. flask with a 3-g. sample. No addition of water is made during the dyeing.

Dyeings by Method IV require an open beaker in order to allow free escape of ammonia, and consequently the volume of the dyebath has to be maintained constant by regular additions of small quantities of hot water.

APPENDIX IV

S.D.C. Grey Scale No. 2

This grey scale is intended to facilitate assessment of the results obtained in the migration test and to place them on the usual numerical basis of five grades. Each step consists of a pair of grey dyeings on wool, and consecutive steps increase in contrast by the same amount. The first pair comprise two identical mid-grey dyeings and are designed to correspond to complete levelling in the migration test; they are therefore given the number 5, in keeping with accepted practice. The second pair, No. 4, comprise one mid-grey and one rather lighter dyeing, thus giving a small contrast. The third pair give greater contrast; and so on down the scale. The highest contrast given by No. 1 pair, is intended to correspond to the lowest degree of migration which would be regarded as commercially useful when using the prescribed test.

The construction of the scale was based on reflection measurements of the grey dyed fabrics in a manner similar to that described in the *Second Report of the Fastness Tests Committee*, Appendix III². The mid-grey dyeing, which forms the starting-point of the scale, has an optical density of 1.04, and each step decreases by an optical density of 0.10 when measured in white light.

APPENDIX V

Constitution of the Committee

The organisations which co-operated in the work of the Committee are listed below—

ORGANISATION	REPRESENTATIVE
Bradford Dyers' Association Ltd.	Mr. E. Wilson
British Cotton and Wool Dyers' Association Ltd. ...	Dr. P. W. Cunliffe
British Hat and Allied Feltmakers' Research Association ...	Dr. T. Barr
Brotherton & Co. Ltd. ...	Mr. J. Starkie
Clayton Aniline Co. Ltd. ...	Mr. J. G. Grundy Mr. B. Kramrisch
John Crossley & Sons Ltd. ...	Mr. G. B. Angus
Geigy Co. Ltd. ...	Mr. H. Briggs
L. B. Holliday & Co. Ltd. ...	Mr. J. Fitton Mr. E. R. Shaw

* These condensers can be made to order by the usual laboratory suppliers.

Imperial Chemical Industries Ltd.	Dr. F. Townend (the late)
	Dr. J. F. Gaunt
	Mr. D. R. Lemin
Sandoz Products Ltd. ...	Mr. G. G. Bradshaw
Wm. Smith & Co. Ltd. ...	Mr. F. Smith
University of Leeds (Department of Colour Chemistry and Dyeing)	Dr. C. B. Stevens
Wool Industries Research Association ...	Dr. F. F. Elsworth
Wolsey Ltd. ...	Mr. J. A. Potter
Yorkshire Dyeware and Chemical Co. Ltd. ...	Mr. A. Thomson

The following also attended meetings of the Committee and took part in its work—

Mr. D. R. Graham	Mr. D. Haigh	Mr. K. A. Hilton
Dr. L. L. Lloyd	Mr. F. Manchester	

Dr. P. W. Cunliffe was Chairman and Dr. F. F. Elsworth Honorary Secretary of the Committee.

The tests described in Appendix VI were carried out under the supervision of Dr. C. B. Stevens.

APPENDIX VI

Classification of Selected Dyes by the Proposed Tests

The dyeings referred to in this appendix were carried out by a technique which was essentially that described in the body of the report but with one modification. To ensure continuous movement of the material through the solution, long strips of white serge sewn to form continuous bands were used. These bands were passed over stainless steel rollers revolving horizontally above the dye beakers, the time taken for one complete revolution of a sample being approx. 20 sec. The results obtained with this modified method were similar to those obtained with the prescribed method.

When a dyeing was completed, the hot solution was immediately decanted through a sintered glass filter to remove loose fibres.

The degree of exhaustion of the dye was determined by colorimetric estimation of the concentration of the residual dye solution with a Duboscq colorimeter and also by producing an exhaust dyeing for comparison with standard dyeings using 7.5% and 12.5% of the original amount of dye taken.

The migrating power of the dye was determined using the full method described in the report, the experiments being carried out in beakers and the solutions boiled gently throughout.

TABLE VII

- (1) The dye named in **heavy type** is that used in the test, the others being its chemical equivalents. Those indicated by asterisks (*) are commercial mixtures and may not be completely identical with one another.
- (2) The results obtained by the optimum dyeing method for each dye are printed in **heavy type**.
- (3) The letter *B* after the dyeing method indicates that the dyeings were carried out in open beakers without using the device described above.

Colour Index No.	Dyes tested and their Chemical Equivalents	Dyeing Method	Exhaustion (%)	Final pH of Dye Liquor	Migration
11	0.88% Kiton Fast Yellow AE (CAC)	I	95.0	3.0	5
	Erio Fast Yellow AEN (Gy)	II	81.1	4.05	5
	Fast Light Yellow E (LBH)				
	Fast Light Yellow E (YDC)				
	Lissamine Fast Yellow AES (ICI)				
	Xylene Fast Yellow ES (S)				
037	0.55% Kiton Yellow S (CAC)	I	89.1	2.95	4.5
	Fast Light Yellow S (YDC)	II	83.9	4.15	5
	Tartar Yellow KS (LBH)				
	Tartrazine KS (ICI)				
	Wool Yellow KS (Gy)				
	Xylene Yellow S (S)				
—	1.1% Polar Yellow 2G (Gy)	II	100	3.9	3.4
	Cloth Fast Yellow GR (CAC)	III	84.7	4.7	4
	Elite Fast Yellow 2G conc. (LBH)	IV	77.0	6.5	4
	Sulfonine Yellow 2G (S)				
—	1.0% Xylene Fast Yellow P (S)	III	92.8	4.8	3.4
		IV	81.0	6.9	3.4
27	1.0% Kiton Fast Orange G (CAC)	I	87.4	3.1	4.5
	Acid Orange GG (Gy)	II	88.7	3.0	4.5
	Fast Light Orange G (LBH)		73.9	4.1	5
	Fast Light Orange G (YDC)				
	Naphthalene Fast Orange 2GS (ICI)				
	Orange G (Br)				
	Xylene Fast Orange G (S)				
151	0.4% Orange II (G.L. J)	I	84.4	3.0	5
	Orange II (Br)	II	82.5	4.1	5
	Orange II (Gy)				
	Orange II (LBH)				
	Orange II (S)				
	Orange II (YDC)				
	Naphthalene Orange GS (ICI)				
161	0.4% Orange R (CAC)	I	92.9	3.05	5
	Acid Orange R (Gy)	II	82.4	4.0	5
	Naphthalene Orange ROS (ICI)				
	Orange R (S)				
	Orange RO (YDC)				
	Orange RO new (LBH)				
—	0.9% Polar Orange R conc. (Gy)	II	100	4.0	1
		III	100	4.7	1
		IV	95.5	6.1	1
		V	90.5	6.8	1
	Cloth Fast Orange 2R (CAC)				
	Elite Fast Orange R conc. (LBH)				
	Sulfonine Orange R (S)				
180	1.3% Brilliant Crimson R (CAC)	II	95.1	3.9	2.3
	Carmoisine LS (ICI)	III	94.8	4.8	3
	Erio Rubine 2B (Gy)	IV	71.0	5.9	3.4
	Omega Chrome Blue F4B (S)				
179	1.5% Brilliant Crimson 2R (CAC)	II	96.1	4.1	3
	Carmoisine (LBH)	III	92.1	4.9	3.4
	Carmoisine (YDC)				
	Carmoisine B (Gy)				
	Carmoisine conc. (S)				
	Carmoisine WS (ICI)				
	Omega Chrome Blue FB (S)				
—	0.85% Brilliant Scarlet R (CAC)	III	90.4	4.8	3
		III	88.3	5.0	3
		III(B)	91.0	4.9	3
	Acid Scarlet 2RE (YDC)				
	Brilliant Ponceau 3R conc. (S)				
	Naphthalene Scarlet BS (ICI)				
	Ponceau 2RE (Gy)				
443	2.0% Coomassie Milling Scarlet GS (ICI)	III	94.5	4.8	1
		IV	92.6	6.3	1
		IV(B)	92.5	6.0	1
	Acid Milling Red G (Gy)				
	Cloth Fast Red GS (CAC)				
	Cyanine Fast Scarlet G (LBH)				
	Milling Scarlet G (YDC)				
	Milling Scarlet 2G (S)				
—	1.4% Kiton Fast Red R (CAC)	I	89.9	3.0	4.5
	Acid Levelling Red 3GS (YDC)	I(B)	90.6	3.1	4.5
	Azo Rhodine GR (S)				
	Erio Floxine 4G (Gy)				
	Lissamine Fast Red 3GS (ICI)				
57	1.1% Kiton Red 6B (CAC)	I	89.4	3.05	4.5
	Acetyl Rose 6BL (LBH)	II	89.2	4.0	5
	Acid Levelling Red 6B (YDC)				
	Azo Rhodine 6B (S)				
	Erio Floxine 6B (Gy)				
	Lissamine Red 6BS (ICI)				
—	1.5% Lissamine Fast Red BS (ICI)	I	98.0	2.9	3
	Acetyl Fast Red B (LBH)	II	85.0	3.8	4
	Azo Rubinol 3GS (S)				
	Erio Fast Floxine BL (Gy)				
	Fast Light Red GL (YDC)				
	Kiton Fast Red BL (CAC)				

Colour Index No.	Dyes tested and their Chemical Equivalents	Dyeing Method	Exhaustion (%)	Final pH of Dye Liquor	Migration	Colour Index No.	Dyes tested and their Chemical Equivalents	Dyeing Method	Exhaustion (%)	Final pH of Dye Liquor	Migration
430	0.9% Polar Red G conc. (Gy) ... Cloth Fast Red GR (CAC) Coomassie Red PGS (ICI) Elite Fast Red G conc. (LBH) Sulfonol Red G (S) Sulphonol Red PG (YDC)	{ II 100 III 100 IV 100	{ 4.0 4.8 6.2	{ 1 1 1		712	1.5% Disulphine Blue VNS (ICI) ... Erio Glaucine supra (Gy) Kition Pure Blue V (CAC) Merantine Blue V (LBH) Patent Blue V (YDC) Xylene Blue VS (S)	{ I (B) II	{ 53.6 24.0	{ 2.8 3.0	{ 5 5
—	1.4% Xylene Fast Red P (S) ...	{ III 98.1 IV 94.6 91.4	{ 4.1 5.0 6.2	{ 2 3 3-4		—	1.4% Polar Brilliant Blue RAW (Gy) ... Brilliant Alizarine Milling Blue BL (S) Cloth Fast Brilliant Blue R (CAC)	{ II 100 III 98.9 IV 86.7 V 37.5	{ 4.1 4.95 6.4 6.4	{ 1 1 1 —	
234	0.25% Resorcline Brown (CAC) ... Naphthalene Leather Brown AS (ICI) Resorcline Brown (S) Resorcline Brown G (Gy) Resorcline Brown pure conc. (YDC) Resorcline Brown R (LBH)	{ II 89.2 III 85.4	{ 4.1 4.0	{ 3-4 4-5		—	1.1% Polar Brilliant Blue GAW (Gy) ... Brilliant Alizarine Milling Blue G (S) Cloth Fast Brilliant Blue G (CAC)	{ IV 100 V 100	{ 6.0 6.6	{ 1 1	
—	1.1% Alizarine Light Blue FPS supra (S)* ... Alizarine Brilliant Blue FPS (LBH)* Erio Fast Cyanine SFP (Gy)*	{ I 91.0 II 91.5 III 90.9 84.8 74.1	{ 3.1 3.0 3.0 4.0 4.8	{ 3 3 3-4 4		—	1.5% Solway Blue PFN (ICI)* ... Alizarine Brilliant Blue PFN (YDC)* Alizarine Sapphire Blue PCF (CAC)* Erio Fast Cyanine CFP (Gy)* Superlan Blue PFN (LBH)*	{ I 91.5 II (B) 84.8 85.6	{ 2.0 3.0 4.0	{ 3-4 4 4	
—	1.7% Alizarine Light Blue 4GL (S) ... Alizarine Light Blue 4GL (YDC) Alizarine Sapphire Blue 4G (CAC) Erio Anthracene Brilliant Blue 4GL (Gy)	{ II 89.2 III 82.0 I 94.1	{ 4.0 4.7 3.1	{ 2-3 3 3		—	1.3% Xylene Fast Blue P (S) ... Alizarine Fast Blue 2BG (CAC) Erio Anthracene Brilliant Blue G (Gy)	{ II 96.5 III 94.5 IV 89.6	{ 4.0 4.7 5.0	{ 1 2 3	
—	1.75% Alizarine Milling Blue SL (S) ...	{ IV 90.5 V 80.5	{ 6.6 6.3	{ 1 1		—	2.0% Lissamine Fast Violet 2BS (ICI) ...	{ I 90.4 II 86.6	{ 3.0 3.95	{ 5 4-5	
—	1.5% Carbolan Blue BS (ICI) ...	{ IV 98.1 V 93.5	{ 6.8 7.1	{ 1 1		—	1.5% Xylene Fast Grey P (S) ... Alizarine Fast Grey P (CAC) Polar Grey G (Gy)	{ III 88.5 IV 78.8	{ 4.8 6.0	{ 2-3 3	
714	1.5% Disulphine Blue ANS (ICI) ... Erio Glaucine XS (Gy) Kition Blue A (CAC) Merantine Blue A (LBH) Patent Blue A (YDC) Xylene Blue AS (S)	{ I 90.0 II 85.0 II (B) 87.5	{ 2.8 3.9 3.9	{ 3 3-4 3-4		References					

- ¹ Report of the Committee on the Dyeing Properties of Direct Dyes, J.S.D.C., 62, 280 (Sept. 1946).
² Trotman, Bell, and Saunderson, J.S.C.I., 53, T 287 (1928);
 Carter and Consden, J. Textile Inst., 37, T 227 (1946).
³ Second Report of the Fastness Tests Committee, Appendix III, J.S.D.C., 64, 144 (April 1948).

LONDON SECTION AND BRADFORD JUNIOR BRANCH

Meetings held in the rooms of the Royal Society, Burlington House, Piccadilly, London W.1, on 7th October 1949, Mr. H. A BRASSARD in the chair; and at the Technical College, Bradford, on 1st February 1950, Mr. G. ARCHER in the chair

The Standardisation of Dyes

R. W. SHACKLETON

The term to *standardise* is one which has changed considerably in meaning since the first world war, and this change in meaning, so far as we are concerned, is a direct result of the rapid progress made by both the dyemaking and dye-using industries.

There is no doubt that the dye famine experienced during the first world war left the impression that standardising a dye was a process designed to mix the maximum amount of common salt with the minimum amount of dye, and that any grinding and mixing operation incidental to this process was merely to disguise the added salt. I have no personal experience of those almost legendary war years of the dye industry, but have gathered the impression that standardisation in those days was very underdeveloped and rather at the mercy of the errors and enthusiasms of infancy.

However, the more stable, and perhaps more exacting, conditions of the early 1920s soon imposed upon the standardising colourist the need to confine his energies to the production of reasonably fine, homogeneous dye powders having tinctorial properties within reasonable commercial limits of the accepted standard. Thus the term to *standardise* acquired a respectable if somewhat prosaic significance, so respectable and prosaic in fact that several people imagine that the job of the standardising colourist makes no appeal to the imagination or challenge to the intellect.

It is one purpose of this lecture to show that this conception of the standardisation of dyes is completely out of date and totally inadequate.

The enormous increase in the variety and classes of dyes and colouring matters now manufactured,

and similar expansions in the uses to which they are put and the methods by which they are applied, have given to the term "to standardise" a meaning so comprehensive and involved that it is not possible in this paper to cover the whole field in any detail. Our attention will therefore be confined to the classification and origin of the types of problems which arise and the methods which can be adopted in the standardising of the ever increasing range of dyes and pigments placed at the disposal of the user.

The problems met with all fall within one or more of the three following classes—

- (1) Control of physical properties
- (2) Control of chemical properties
- (3) Control of tinctorial properties.

The control of tinctorial properties is, of course, largely dependent on the control of physical and chemical properties, but there are aspects of this problem deserving special mention.

1. CONTROL OF PHYSICAL PROPERTIES

One of the significant changes of the last few years in both the dyemaking and dye-using industries has been the ever growing importance of control of physical properties. The ever expanding variety of materials to which colour, in one form or another, is applied, and the increasing mechanisation of application processes, increase the demand for colouring matters which will, first of all, lend themselves to mechanical processing, without damage to the machine, and which can be used either in a continuous process, as in bulk production, or in a series of short runs at infrequent intervals, and yet produce a standard result on each occasion.

If the dye is applied from solution, then in all cases freedom from insoluble matter is essential and must be controlled; e.g. the presence of relatively small amounts of insoluble matter in a dye used for cop dyeing can cause considerable embarrassment to the dyer, while the presence of relatively few black specks of insoluble matter in a solution of, say, Edicol Tartrazine NNS by no means encourages its use in the colouring of foodstuffs.

The presence of iron rust, siliceous matter, and other types of hard gritty substances can cause scratching of costly engraved printing rollers and give rise to a surprising number and variety of serious troubles.

In some industries, e.g. the foodstuff-colouring and textile-printing industries, it is common practice to use concentrated solutions of dyes and, in some cases, to keep these solutions for relatively long periods, so that solubility, stability of solutions, and in some cases storage properties must be kept under control.

In supplying a world market, too, the question of solubility becomes even more complicated by the fact that the quality of solvents varies considerably; thus it is not uncommon to use water of 10–15° hardness in dyeing processes.

Whether the dye is dispersed for use in a dyeing operation, a coating composition for leather, paper, or textile fabric, pigmentation of a latex or spinning

solution, or the production of the wide variety of water and oil paints, particle size, wetting-out properties, dispersibility, aggregation, and stability of dispersion must be carefully controlled.

2. CONTROL OF CHEMICAL PROPERTIES

It is well known that relatively few dyes manufactured for commercial usage are pure chemical compounds. Impurities and isomers are present or are formed, in raw materials, primaries, intermediates, and dye-manufacturing stages, and for the purpose of the standardising colourist the constitution of a dye, so simply quoted in the *Colour Index*, is of less importance than a knowledge of the kind, amount, and effect of the other organic and inorganic compounds which may be present in the batch material he is called upon to standardise. Thus chromatographic and chemical analysis of Chlorazol Black E will reveal that this dye is actually a mixture of at least six, and possibly more, coloured organic compounds, together with some sodium chloride, sodium sulphate, and sodium carbonate. This extreme case is quoted only to emphasise that in many instances the standardising colourist is called upon to control the behaviour of a complicated mixture of compounds having different tinctorial properties.

The fact that many dyes are, in effect, mixtures does not detract from their commercial utility; in fact the effects produced by many of these mixtures, manufactured over a number of years, have become so established that the absence of the impurities would render the dyes less attractive to the user. For example, it is obvious that in the early days of the manufacture of γ -acid, in this country and others, this intermediate contained approx. 5% of the isomeric J-acid, since it is impossible to achieve the accepted hue of a dye such as Chlorazol Violet N by using pure γ -acid. In this particular case the presence of J-acid confers redness and brightness.

The presence of these "impurities"—for lack of a better term—gives rise to trouble only when the kind and amounts of impurity vary from batch to batch. Thus one of the impurities present in very small amounts in Chlorazol Black E is a bluish-red dye having no affinity for cellulose, so that in normal textile usage the impurity remains in the dyebath. When used in dyeing chrome calf skin the impurity is so soluble that it penetrates the leather, and if large amounts were present the characteristic green cross cut of a chrome calf would be replaced by a dull grey.

Vat oranges can be produced by bromination of certain anthraquinone compounds, and the resulting dye may be a mixture of mono-, di-, tri-, and polybromo compounds. The different bromine compounds which go to make up the commercial dye are very similar in hue, but will certainly vary in solubility and sensitivity to hard water, so that it is possible to obtain mixtures having the same shade and strength under one set of dyeing conditions but giving wide variations under other conditions.

It will be appreciated then that, while the standardising colourist cannot effect any fundamental change in the chemical constitution of the

batches he is required to standardise, he can correct some undesirable abnormalities and can, by suitable blending of batches, balance the constitution and properties. Far more important, as will be explained later, he should play his part in standardising the dye even before he receives it from the manufacturing department.

3. CONTROL OF TINCTORIAL PROPERTIES

It will already be clear that in the process of standardising a dye the control of tinctorial properties is not merely a question of adjusting to a given shade and strength. Any control or adjustment of these properties must be made with due consideration of all the uses to which the product is put and to any other effects which may be produced by materials or processes used to correct shade or strength.

A number of dyes are required to colour a variety of products and yet not cause coloration of other materials which may be present during application of the dye or in subsequent processing. Thus certain dyes used for the dyeing of cotton and viscose are required to reserve acetate rayon, and if used for burl dyeing should not stain wool. Certain dyes for wool, in their turn, are required to reserve cotton and viscose, and many dyed textiles when washed must not stain adjacent whites.

In the pigments field, too, bleeding into solvents on transparent surface coatings or marking off by contact with other materials can be caused by impurities, present in small amounts. It is suggested, therefore, that while the chief methods of control of these factors depend on control during manufacture, the standardising process must include some degree of tinctorial control of resist effects, degree and type of stains, bleeding into solvents, and marking-off properties, and any adjustment made with a view to correcting shade and strength must take into consideration all the secondary effects.

In a similar manner it will be known that the shade and strength of a pigment to be dispersed in a liquid medium can be modified by variation of the physical condition of the pigment, but modifications carried out with the object of bringing shade or strength to standard must not result in a product having an abnormal physical form.

TECHNIQUE OF STANDARDISATION

In standardising the crude batch of dye received from the manufacturing department, which may represent the result of a large number of intricate chemical processes, there are three essential operations—

(1) **ESTABLISHMENT OF A STANDARD**—The standard is, or should be, tangible evidence of the product being offered for sale, and the customer has the right to expect that any deliveries made, during the current life of the standard, should be commercially equal to this standard.

(2) **ASSESSMENT OF BATCH MATERIAL PRIOR TO FINAL STANDARDISING OPERATION**—It is axiomatic, of course, that before batch material can be converted to the finally standardised material it must be compared and measured against the selling

standard. It is less obvious, perhaps, that it is at this preliminary stage that standardisation far more fundamental and important than the so called final standardising operation can be carried out.

Here the standardising colourist can provide a batch-to-batch running commentary on the quality of manufacture and can bring to light batch-to-batch variations in physical, chemical, or tinctorial properties which have undesirable effects which cannot be modified or eliminated in the final standardising process—in other words, he is constantly indicating to the experimental and manufacturing chemists the instabilities and inaccuracies of the manufacturing process which produce the impurities, or variation in impurities, so liable to cause difficulties. He seeks out the evidence of lack of manufacturing control, and in so doing extends the scope of the standardising operation from the first stage of manufacture to the final operation. It is at this stage that material not considered suitable for conversion to the sales product is rejected.

(3) **CONVERSION OF BATCH MATERIAL TO SALEABLE DYE (FINAL STANDARDISING OPERATION)**—Obviously it is essential that, both in the assessment of batch material and in the final standardising operation, criteria should be established to determine whether a batch or mixing is equal to standard under all conditions of usage to which it is put. It can be and has been argued that, as the value of a dye or pigment depends on the visual effects it produces, the criteria should be based on tests carried out under actual, or as nearly as possible actual, user conditions. However, such a state of affairs is neither possible nor necessary. Obviously it is necessary to have one or more tests which reveal tinctorial properties such as shade, strength, absence of staining, etc.; but if physical and chemical properties can be standardised and controlled, it is reasonable to assume that a sample will behave as standard under all conditions of use.

By experience and test it is possible to define conditions under which a dye will meet all known requirements. Thus if a sample of Solochrome Brown RS is equal to standard when dyed on wool, it will be equal to standard when dyed on skiver if the sample is equal in solubility and leaves no abnormal exhaust liquor in the wool dyebath. It will also produce the same result on anodised aluminium if the pH of a 1% solution is equal to that of a 1% solution of standard.

Similarly, a sample of Chlorazol Black E will dye a variety of textile fibres, paper, leather, etc. equal to standard if it is equal to standard when tested on cotton, and has equal solubility, alkalinity, affinity, and exhaustion properties. The standardising colourist, therefore, is constantly seeking tests which will reveal when a sample is different in some way from his standard; and when such abnormalities are revealed, he must then translate the abnormality into terms of effect either by his experience or by effect tests.

The demand for suitable instruments has been, and is being, met by instrument makers, so that the standardising colourist has many instruments in

daily use which were not available twenty or thirty years ago, e.g. pH-meters, colorimeters, spectrophotometers, and absorptiometers. The ability to measure quickly and accurately the optical density of dye solutions, dyebaths, etc. provides not only a method of increasing the accuracy of strength estimations but also a means of assessing more fundamental properties. In the near future instruments such as the Hardy self-recording spectrophotometer will become invaluable aids to still more accurate control of both manufacture and standardisation. An instrument such as the Hardy, which provides accurate repeatable recordings of transmission curves of coloured solutions or reflection curves from a variety of coloured surfaces, opens up a new and rather exciting vista to the standardising colourist.

In other fields the use of instruments for measuring the physical properties of dyes and pigments, such as particle size, dispersion, sedimentation, texture, etc., is increasing our knowledge of their fundamental properties and control.

No less important are the attempts being made to improve the accuracy of laboratory tests based on methods of application used in industry.

Years ago one heard arguments that, as the final assessment of the effects produced by dyes and pigments depended on the human eye, any methods of test which were equal in accuracy to the human eye were satisfactory. It is true, of course, that the trained eye of the experienced standardising colourist provides a quick method of assessing and comparing coloured patterns, and it is true, also, or at least we like to think so, that the true colourist has something of the artist in his make-up and craft, but it is far more satisfactory to have even an expert human judgment confirmed by an accurate instrument. It is far more important that the instrument can describe and record in physical terms differences which, when examined by the human eye, are not readily described or understandable to those who have not seen the actual test.

Scientific control of chemical processes of manufacture and of the finished standardised product cannot be attained unless the criteria are scientific and accurate. For this reason the need to carry out laboratory dye tests and other application tests with analytical accuracy and repeatability is increasingly recognised, the Marney

dyeing machine being one example of recent progress.

Many developments are the direct results of a demand created by a new and specific method of use, so that the customer can, and does, provide a strong incentive to progress, this incentive being reinforced by the fact that efficient and scientific control of a manufacturing process calls for similar exacting control.

When the standardising colourist issues a mixing for sale which, because of lack of control of strength, is either 5% weaker or 5% stronger than standard, the efficient manufacturing chemist wishes to know the reason why he gets differences of 10% yield between one batch or series of batches and another, and can waste considerable time, effort, and money looking for a variation in his process which does not exist.

If the physical characteristics of the batch material and the requirements of the finished material are known and can be specified, methods required to convert the batch to the finished material can be determined. Similarly, the question of additions made during the standardising process is not merely one of correcting shade and strength. If a diluent is added its suitability for the purposes for which the dye is used will be considered and its effect on solubility, etc. taken into account.

The colourist has available adjuvants for the correction of acidity or alkalinity, the improvement of solubility, wetting-out properties, or dispersion, or for countering the effect of hard water, etc. When the shade of manufacture shows variation from standard, he will first of all eliminate variations as far as possible by suitable blending of batches, and any shading dye that he adds will be chosen because it has properties similar to those of the dye to which it is added.

Always he aims at the manufacture and standardisation of a product which will behave exactly as standard under all conditions. This is not always possible, of course, since the requirements of one method of usage may be entirely different from those of another, and in such cases he must produce from the same batch material special qualities to meet special requirements.

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SCOTTISH SECTION

Lecture given in the Refresher Course on Textile Science and Technology at the Royal Technical College, Glasgow, on 20th November 1948, Professor W. M. CUMMING in the chair

Finishing Machinery—Principles and Development

K. S. LAURIE

Introduction

In this lecture we are concerned with the principles underlying the design and development of cloth-finishing machinery. It is not possible to cover the whole ground of finishing machines, and, therefore, the examples will be limited to quite a small range, which will, however, suffice to show some of the underlying considerations. It is necessary to take into account the chemical, physical, and mechanical aspects of machine design.

1. CHEMICAL ASPECTS OF MACHINE DESIGN

All parts of finishing machines must be constructed of materials that will withstand the chemical conditions prevailing in the particular operation. These conditions include resistance to various liquors used in cloth treatment, resistance to the action of the cloth itself, and also resistance to the atmospheric conditions prevailing in the finishing works. The last item can be a particularly severe tester of materials. Not only material but also shape and method of construction must conform to the chemical requirements; thus we find that in dyeing machinery the tanks or boxes are made of stainless steel, with well rounded corners, and of dimensions as nearly as possible suitable for the best chemical conditions.

2. PHYSICAL ASPECTS OF MACHINE DESIGN

Cloth during finishing processes has to be subjected to the physical actions of—

- Liquids
- Pressure
- Heating by conduction, convection, and radiation.

These involve the study of liquid flow, of roller pressure, and of the transfer of heat from hot surfaces and from hot gases.

3. MECHANICAL ASPECTS OF MACHINE DESIGN

Cloth to the engineer is a peculiar material, differing from most of the other materials which he commonly has to handle. From the mechanical point of view it has the following disadvantages—

- Little or no stiffness. Cloth can be pulled but cannot be pushed, and this feature introduces difficulties in mechanical handling.
- Very irregular dimensions, particularly in width.
- Extreme variety of construction, material, and weight.
- High cost or value.

Let us see how we deal with these considerations in practice. We shall examine the two important types of process, dyeing and the subsequent drying of cloth.

Dyeing Machines

The following features are generally considered to be required in the ideal dyeing machine—

- Small liquor : cloth ratio. This produces the best chemical conditions for dye exhaustion, and allows the economical use of salts.
- Open-width processing where possible.
- Minimum cloth tension.
- High speed of operation.
- Economy in heat.

The types of dyeing machine most commonly in use are jiggers, wince machines, and padding mangles.

JIGGERS

These machines are commonly made with a small stainless steel box and satisfy fairly well conditions (a) and (b). Condition (c) is not well fulfilled, as jiggers inevitably require a considerable cloth tension for successful functioning. A good design of spreader or expanding roller will reduce the cloth tension considerably, since the minimum tension required is that which is sufficient to operate the spreader and so eliminate creases. Even with the best spreaders, however, the cloth tension is still far too high for cloths such as woollen and spun-rayon fabrics and all types of crêpes and knitted goods. The speed of processing (d) is largely dependent on the rate of levelling, but the actual loading and unloading of cloth is fairly rapid. There are several special designs of jigger which reduce the cloth tension and improve the evenness of dyeing to some extent, besides adding automatic handling features. An outstanding example is the Swiss Benninger jig, which has been well tried over a number of years and which undoubtedly extends the use of jig dyeing to fabrics which could not otherwise be processed in this way.

WINCE (WINCH) MACHINES

Wince machines still have to be used for a wide range of cloths where tension is not desirable. It is possible to have a fairly small liquor : goods ratio, even as low as 5 : 1 when the machine is fully loaded with cloth. Usually, however, it is desirable to work with less cloth than would be required for the minimum liquor ratio. The outstanding virtue of the wince machine is the low tension applied to the cloth, and because of this a number of disadvantages are accepted. Wince machines are slow to unload and load, requiring considerable labour, and have large volumes of liquor to change, to heat up, and to discharge; the heat losses are considerable and the operating temperatures uneven.

Wince machines should be designed for easy flow and folding of the cloth, allowing a kind of opening action as the cloth glides through the dyebath.

Their means of heating should be as rapid and even as possible without causing too much strain on the boiler plant. The wince should be made to give minimum cloth tension but also no slip. This in practice is rather difficult to achieve with the modern stainless steel construction. Freedom from cloth entanglement is another object of design, involving attention to the shape of the box and the construction of the peg-rail. In this respect, different materials require different shapes of dye-box. Heavy wool and knitted goods are best processed in deep boxes, while light silk and rayon crêpes require comparatively long and shallow dye-baths. Modern boxes aim at conserving heat and attaining even bath temperatures by means of hoods covering the whole machine. This has to be done with careful design, so that complete enclosure is combined with ease of operation and of loading and unloading.

PADDING MANGLES

Dyeing processes carried out in padding mangles are different in principle from those done in a dye-bath. The cloth has now to pick up a continuous small amount of dye liquor, and there is no question of allowing time for levelling the shade. In many cases, particularly with pigment padding, the impregnating liquor does not contain any substance which has an affinity for the fibre. It is therefore essential that the design of padding mangles should allow a very even pick-up of liquor, controllable in quantity. This is achieved by providing bowls of adequate size and strength, with good and durable coverings, supported in a strong frame. The most convenient form of pressure application is by means of compressed-air cylinders controlled by a reducing valve. It is usual to use a very small liquor trough, supplied from a storage tank of adequate size.

Production by pad dyeing is a relatively rapid process, since it is continuous and no time has to be allowed for levelling. When producing pigment pads, however, the impregnated cloth has usually to be reduced and subsequently oxidised. These operations are frequently carried out in jiggers, thus involving the usual loss of time due to a batch process. Later developments, particularly those due to American technologists, allow the reduction and oxidation to be carried out continuously. The best known of these processes, the Du Pont pad-steam process, involves the following operations—

- (a) Pad with unreduced pigment.
- (b) Dry in roller-type drier.
- (c) Pad through a hydrosulphite and caustic reducing liquor.
- (d) Pass immediately into a steaming chamber consisting of a short roller-type machine.
- (e) Soap and wash.

This process is exceedingly rapid, particularly the effect of the steam after reduction padding. The quantities of cloth required to make the process commercially useful are rather too large for British and Continental practice, but the system appears to have great possibilities in extending the use of the pad-mangle method of dyeing. It introduces a new set of engineering problems, particularly in

the design of suitable drying and steaming machines.

Principles of Drying

Removing water from fabric has to be done on more than one occasion during the dyeing and finishing process. Unfortunately it is one of the most expensive operations in finishing and requires the most expensive plant to carry it out. As an example, stenter room plant will occupy approximately four times the space and will cost nearly four times as much as the plant required for dyeing the same quantity of cloth in the same time. Stentering is, of course, only one method of drying and is probably the most expensive, but its use is becoming almost universal, especially for final finishing. In many works it is found that shortage in drying plant has to be overcome by working the drying section of the works on a double shift.

MECHANICAL REMOVAL OF MOISTURE

It is well worth emphasising the great saving in cost and energy shown by mechanical methods of removing water as against methods which require heat. As an approximate example, cloth entering a mangle with 200% moisture will be reduced to a moisture content of 60%. Thus the mangle will remove 140%, amounting, say, to 900 lb./hr. for an energy consumption of about 6 kw.-hr. To remove 900 lb. of water by heat and evaporation would require about 600 kw.-hr. or 100 times as much. It is therefore essential to remove as much moisture as possible by mechanical means. With this in view machinery makers are developing heavier and more efficient mangles and are paying attention to the design of suction extractors and centrifugal extractors. So far as is known at present an efficient mangle should possess the following features—

- (a) Large-diameter bowls to give even squeezing without undue deflection.
- (b) Suitable covering, usually a hard nickel face on the metal bowl and a substantial rubber cover of about 60° P. & J. hardness.
- (c) Suitable pressure system, easily operated, and sufficiently powerful for the maximum extraction.
- (d) Drive of adequate power, so arranged as not to influence the pressure on the bowls.
- (e) All parts to be made from material suitable for the chemical conditions.

The physical factors governing the performance of mangles are not yet well understood, and a programme of research is being undertaken on this subject.

LIMITS OF MECHANICAL DRYING

Even in the most favourable cases the moisture removed by mangles leaves a considerable residue in the cloth. The following are common percentages for the moisture left in fabrics after mechanical removal. They are based on the dry weight of the fabric.

Cotton	70-90
Rayon	70-100
Spun rayon	80
Wool	45-60
Nylon	30

The above percentages of moisture remaining represent the practical limit to which mechanical extraction of moisture can be taken. It might be possible, by the use of extreme pressures or extremely high extractor velocities, to reduce this percentage, but even with the great difference in cost between mechanical and thermal methods of moisture extraction it might not be economical to do so. There is also a limit imposed by the liability of cloth to mechanical damage. Therefore, in practice the above residues can be extracted only by the application of heat in various ways.

DRYING BY HEAT

The removal of moisture from fabrics by the application of heat depends considerably on the efficiency of heat transfer from the heating medium to the textile, and conditions which favour a high rate of transfer also favour a high rate of drying. High rates of drying are economically important owing to the resultant saving in floor space and in labour costs. In addition, however, to a high rate of heat transference, it is necessary to provide sufficient air to absorb the moisture liberated, this being the only practical way in which the moisture can be carried away.

The general conditions, therefore, for drying are—

- (a) Sufficient heat must be transferred to the moisture to raise its temperature and to provide the necessary heat of evaporation.
- (b) Sufficient air of low moisture content must be supplied to absorb the moisture evaporated.

Heat for drying can be transferred to the moist cloth by conduction, convection, or radiation, and most drying methods are a combination of two or more of these. We shall confine our considerations to air drying, which is mainly a convection method, although most air drying machines employ incidentally quite a large proportion of radiated heat.

CONDITIONS FOR EFFICIENT AIR DRYING

Convection drying covers all the air drying processes used in the textile trade, although there is generally some assistance from radiant heat.

The two conditions given above—sufficient heat transfer and sufficient absorbing air—are still the main requirements. The first requires that the air, the heat conveyor, should pass the cloth at high speed to ensure efficient transfer of heat to the moisture. The second condition requires that the air passing thus rapidly shall be in a state to receive the moisture readily. Relative humidity must be kept low. There must be economic compromise in both these conditions. The fan horse power required to produce high air speeds rises much more rapidly than the drying effect, and the presenting of very dry air to the cloth may represent an undue waste of steam. The air temperature has a large bearing on the rate of output: the higher the temperature the more rapid the heat transfer. It is a mistake, however, to associate high air temperature with high cloth temperatures. In theory the temperature of the moisture being evaporated is the same as the *wet-bulb temperature*

of the drying air. If the drying air has a low humidity the moisture temperature, and therefore the cloth temperature, remain low so long as there is moisture present in the cloth. The cloth does not begin to get really warm until it is dry—by which time it should be coming out of the machine.

EVENNESS OF DRYING

It is well to stress the necessity of *even* drying effects. If parts of the cloth are dry before the rest there is risk of these parts being overheated before the cloth is completely dry.

It can be seen that a good modern air drying machine should aim at high air temperatures, low relative humidity, and even drying, all without undue fan horse power and steam consumption.

Nearly all modern drying machine systems, both for stenters and for other types of drying, employ the cross-jet type of air drying. This was originally developed in this country by Spooner, but has since been modified to allow easier access and an easier flow of air. By such modifications one design in particular is enabled to employ high-efficiency axial-flow fans blowing from both sides of the machine, supplying top and bottom air chambers with an ample volume of air. The steam heaters in this design are large and easily accessible, the air passages are also ample in cross-section, and these features enable a high rate of drying to be carried out with a very low fan horse power.

THE AIR-LAY DRYER

A modern type of cloth drier, in which the above principles are utilised, is known as the Multipass Air-lay. A chain conveyor, the cross members of which are spaced cork-covered bars, takes an up-and-down path through an insulated chamber. Air, circulated by fans, passes through steam heaters and emerges at high speed through slots directed against the face of the conveyor. The cloth is fed on to the conveyor and is held there by force of the drying air issuing from the slots. This machine has the advantages of avoiding cloth strain, allowing full shrinkage while drying, requiring small floor space in relation to output, and, owing to the efficient drying conditions, short period of cloth in the drying chamber.

STENTERS

The ordinary automatic clip stentering machine is well known and it is not intended to describe it fully here. Apart from the heating system, the modern improvements to stenters involve mainly better provision for housing the driving parts. Totally enclosed gear-boxes with adequate lubrication are now standard features, and driving by means of high-speed splined shafts follows modern engineering practice.

PIN STENTERS

It is worth while, however, drawing attention to the development of the modern pin stenter machine. This was the old original type of stenter, but since the introduction of rayon finishing it has been revived and modernised. Its main purpose is to allow warp shrinkage while drying, a feature which does not exist on the clip machine. This

shrinkage is carried out by means of an overfeed gear, which in its present form is a German invention.

Various problems have had to be overcome during the engineering development of the pin stenter. Even after a successful overfeed gear was established, the problem of accurate automatic guiding of the cloth entering the machine had to be solved. The modern hydraulic guider, although not perfect, has gone a great way towards the complete solution of this problem. An electronic guider, now in course of development, promises well for further progress, especially for the high-speed running of difficult cloths. At the present stage the limitation on the production speed of pin stenters is still the guiding of certain types of difficult light-weight fabric. When this problem is completely solved it will be possible to produce much faster running stenters, equipped with predrying machines, and so to increase considerably the economic value of drying plant for silk and rayon.

In applying hot air to pin stenters it is especially necessary to have a proper balance between the air streams above and below the cloth, on account of the nature of the cloths which are frequently processed on pin stenters. Certain crêpe goods have to be finished with a large amount of overfeed and with very little weft tension, and unbalanced air streams are liable either to blow the cloth off the pins or to produce undue stretching in the width.

INFRA-RED DRYING

The application of radiant heat to cloth drying has been considered to some extent lately, and we should discuss briefly the implications of this. Most infra-red applications are in the form of electric filament lamps with suitable reflectors. It must be remembered as a fundamental of drying that sufficient heat must be provided to evaporate the water. When this heat is provided in the form of electricity it is under present conditions relatively expensive—say six times the cost of supplying it by steam.

Some experiments were carried out on drying fabrics by means of a battery of infra-red lamps. The efficiency of the operation was found to be under 30% for a dark cloth and under 20% for a white cloth. Steam drying efficiency in air drying machines approaches 50%. The conclusion, therefore, is that infra-red drying is likely to cost 10–15 times as much as steam-air drying. A drying machine evaporating, say, 600 lb. of moisture per hour would require approx. 1200 lb. of steam, or, if infra-red heated, 870 kw. of electrical power.

Electricity is not, of course, the only source of infra-red rays. Gas-heated elements have the advantage of lower running cost and far greater heat concentrations, though it is doubtful whether their efficiency of application is any greater.

Infra-red rays can also be used for heating alone, as distinct from drying, and this application may have possibilities in the curing of plastic cloth finishes and similar applications.

Application of Electronics

Electronic means are now in use in one or two applications in the control of finishing machinery.

CLOTH GUIDING

It has been mentioned already what an important problem the correct guiding of cloth into pin stenters has become. One of the more promising solutions of this has been by means of an electronically controlled guide wheel at the entrance to the stenter rail. The position of the cloth selvage is detected by means of a beam of light reflected on to a photosensitive cell and partly obscured by the edge of the cloth. The voltage thus generated is amplified and finally applied to a small servo-motor, which changes the angle of the guide wheel to follow changes in position of the cloth edge.

MOISTURE DETECTION AND CONTROL

This is a wide subject of great interest, and there is only space here to deal with it very shortly. There are several methods of detecting electrically the amount of moisture present in cloth which is nearly dry, but of these only two are of interest at the moment.

Resistance Method

One of these methods is to determine the electrical resistance of damp cloth and to measure the changes in resistance which take place with changing moisture content. The resistances involved are frequently of a very high order, approaching thousands of megohms. The electrical indications received are therefore very small, and require electronic amplification. The resultant current or voltage can be read on a meter or can be used to operate a machine control. This is the basis of the Shirley hygrometer, and also of the American Brown Instrument Co. instruments. The resistance method gives very reliable indications and the main difficulties are those of calibration.

Capacity Methods

Another method of moisture detection in fabrics is to treat the moist fabric as a dielectric between the plates of an electrical condenser. The presence of moisture alters the dielectric constant of the fabric and thus produces a change in current, which can be amplified and employed in the same way as in the resistance method.

MOISTURE CONTROL

The moisture content of cloth, after it has been detected and measured, can be controlled in various ways. The most usual is to control the speed of operation of the drying machine producing the fabric, so that the fabric leaves the machine with just the correct amount of moisture in it. From the engineering point of view, this is not a simple control problem. When an alteration is made to the speed of a drying machine in response to an error in moisture content, the effect of the speed alteration is not completed for a considerable time. This time lag is much longer than those usually encountered in control problems. The method commonly employed to overcome this difficulty is to sample the moisture content at intervals corresponding to the time taken for the cloth to travel through the drying machine. The speed-correcting motion is then made only at these intervals. This method

has the obvious disadvantage that moisture errors may take place between the times of correction, but in practice it has proved to be reasonably satisfactory. It would theoretically be possible to operate a continuous system of control, but in practice, owing mainly to the great variety of cloth construction and weight, the difficulties are too great.

When using the intermittent or sampling method, it is important that the amount of speed correction bears some relation to the moisture content error which gave rise to it. This is accomplished in one electronic control system by allowing the indication of moisture content obtained electrically from the measuring instrument to charge a condenser, the charge of which subsequently leaks away at a rate which can be adjusted. So long as the voltage of the condenser remains above a certain level the speed-operating gear will continue to make a change in the speed of the machine. Thus the amount of speed change permitted depends on the

amount of the moisture error. The speed-changing apparatus is in this case set in motion by means of a thyatron valve, which can pass current only when the condenser voltage applied to its grid remains above a given level.

Conclusion

In the course of a lecture such as this it has been possible to deal with only a few of the problems connected with finishing machinery. It is hoped, however, that enough has been said to show the principles on which such machinery should be developed. A great deal has yet to be learned with regard to these principles, and it is only by co-operation, both with scientific bodies and with the textile-finishing trade itself, that the textile engineer can make progress in this important branch of machinery. It is good to know that this co-operation is undoubtedly taking place, and that advances in design and construction are consequently being made.

COMMUNICATION

A Review of the Various Methods extant for the Preparation of Aromatic Nitro Compounds, excluding those of Direct Nitration

H. H. HODGSON, F. HEYWORTH, and E. R. WARD

INTRODUCTION

The most common process for preparing aromatic nitro compounds is that of direct nitration, in which the compound to be nitrated is treated with nitric acid, either alone or mixed with concentrated sulphuric acid, acetic acid, or acetic acid and anhydride. Such direct nitration, however, is not of universal application, some of its most pronounced limitations being—

(1) The directing groups in the compound to be nitrated may permit only a few of the possible isomers to be formed. Such limitation is of more significance in the naphthalene than in the benzene series, since the possible number of isomers is much greater in the former; thus only two of the seven possible dinitronaphthalenes can be obtained by the direct nitration of α -nitronaphthalene, viz. the 1:5- and 1:8-isomerides¹.

(2) Mixtures of isomers are invariably produced; e.g. the mononitration of naphthalene itself affords 96% of α - and 4% of β -nitronaphthalene².

(3) Polynitration may not be preventable if the compound for nitration is very reactive, while the same applies when severe nitration conditions have to be employed to initiate nitration.

(4) Variations due to solvent effect may determine the facility of nitration and so afford a complex mixture of products; e.g. naphthalenedisulphonic acids give dinitration products with 90% sulphuric acid, but only mononitration products with the 100% acid³.

(5) By-products invariably arise from concurrent oxidation and nitration (oxidation being generally favoured by temperature rise and excess of nitrating acid); or products are formed through the influence of the sulphuric acid if present, and

such, being usually more soluble in the nitrating acid than the nitration product desired, do not separate but remain to contaminate the final product when it is precipitated by dilution or by pouring the nitration mixture on to ice. Such by-products are usually very difficult to remove. Lantz³ found that the mononitro derivatives of 1:5- and 1:6-naphthalenedisulphonic acids were transformed in 100% sulphuric acid, at about 60°C., giving probably nitrosonaphtholsulphonic acids, which were not further nitrated, whereas the 2:6- and 2:7-isomerides were dinitrated under the same conditions.

INDIRECT METHODS FOR THE PREPARATION OF AROMATIC NITRO COMPOUNDS

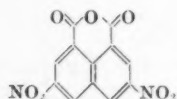
In order to overcome the limitations of direct nitration, various indirect approaches have been devised, and such methods are essential in naphthalene chemistry, where the rarer polynitro compounds can only be prepared indirectly. These methods are—

(1) DIAZOTISATION AND DEAMINATION OF NITRO-AMINES—This approach has been most useful for obtaining the rarer dinitronaphthalenes. The most convenient procedures are those of Hodgson and Turner⁴⁻⁶, whereby the amine is diazotised by the nitrosylsulphuric-glacial acetic acid process of Hodgson and Walker⁷, and the solution of the diazonium sulphate added to a suspension of cuprous oxide in ethanol, or better, methanol⁸; and of Hodgson and Birtwell⁹, where the amine, diazotised as before, is deaminated by addition of cuprous oxide to the diazonium solution. These methods readily afford β -nitronaphthalene from 2-nitro-1-naphthylamine in 80% yield; and the

1:3-, 1:4-, 2:3-, and 1:6-dinitronaphthalenes from 2:4-dinitro-1-naphthylamine, 1:4-dinitro-2-naphthylamine, 2:3-dinitro-1-naphthylamine, and 1:6-dinitro-2-naphthylamine respectively. Where the dinitronaphthylamine arises from the nitration of a *p*-toluenesulphonamido derivative, then the nitrated derivative can be directly hydrolysed, diazotised, and deaminated without isolation of intermediate products⁹.

Anomalous results, however, have been obtained in some cases: thus, Hodgson and Hathway¹⁰ found that, in the deamination of 1:4:6-trinitro-*p*-toluenesulphon-2-naphthalide in methyl alcohol with cuprous oxide, there was formed 4:6-dinitro-1-naphthol instead of the anticipated trinitronaphthalene. An excellent review of this deamination reaction is given in *Organic Reactions*¹¹.

(2) DECARBOXYLATION OF NITROCARBOXYLIC ACIDS—One of the available methods¹² for the preparation of the rare 2:7-dinitronaphthalene consists in nitration of naphthalic anhydride followed by decarboxylation of the dinitro product—



by means of copper bronze in quinoline. Other examples are the nitration of 8-nitronaphthalene-1-carboxylic acid¹³ and of 5-nitronaphthalene-1-carboxylic acid¹⁴, to give 1:8- and 1:5-dinitronaphthalene respectively.

Incidentally, mention may be made here of the dechlorination of chloronitrohydrocarbons by copper powder, a method used to prepare 1:3:8-trinitronaphthalene from 4-chloro-1:3:8-trinitronaphthalene in nitrobenzene as solvent. Similarly, 1:3-dinitronaphthalene has been obtained from 4-chloro-1:3-dinitronaphthalene¹⁵.

(3) OXIDATION OF OXIMES AND NITROSO AND AMINO COMPOUNDS—This method has been of limited application. The oxidising agents employed include Caro's acid¹⁶, ammonium persulphate with silver nitrate as catalyst¹⁷, peracetic acid¹⁸, aqueous sodium peroxide¹⁹, and fuming nitric acid²⁰. The most common application has been the preparation of *p*-dinitrobenzene from *p*-benzoquinone dioxime²⁰ and of 2:5-dinitrotoluene from 5-nitro-*o*-toluidine²¹.

(4) ELIMINATION OF HYDROGEN HALIDES—1:2- and 1:3-Dinitronaphthalenes have been obtained from the 1:2- and 1:3-dinitrotetralins by bromination to the respective dibromotetralins, which, when heated, evolve hydrogen bromide to form the respective dinitronaphthalenes²².

(5) REPLACEMENT OF THE DIAZO BY THE NITRO GROUP—This method was first introduced by Sandmeyer²³ in 1887, and has the advantage over previous processes that the number of nitro groups is increased, i.e. a dinitronaphthalene can be obtained from a mononitronaphthylamine. Sandmeyer's original procedure was to diazotise the amine in nitric acid solution and add this to a solution of sodium nitrite containing cuprous oxide as catalyst; in general, poor and uncertain yields

were obtained, e.g. 5% of β -nitronaphthalene from β -naphthylamine.

Modifications by later workers include the use of cupro-cupri sulphite as catalyst²⁴, the red variety known as Chevreul's salt being more efficacious than the white Etard's salt. This catalyst is prepared by adding an equimolecular quantity of copper sulphate in aqueous solution to one of sodium sulphite, filtering, and washing the precipitate to remove any sulphite that might not have reacted. With this catalyst the yield of β -nitronaphthalene from β -naphthylamine was increased to 25%. Meisenheimer and Pätzig²⁵ applied the replacement method to the preparation of nitrotoluenes, using cuprous oxide as catalyst and excess of sodium nitrite.

By adding twenty molecular proportions of nitrite to 2:4:6-tribromobenzenediazonium sulphate in dilute solution, Orton²⁶ obtained 2:4:6-tribromonitrobenzene in 80% yield. In this case the important observation was made that no catalyst was required.

Copper bronze with sodium nitrite was used by Veselý and Dvořák²⁷; also by Bucherer and van der Recke²⁸. In every case, however, the reactions were of very limited applicability.

Contardi^{29,30} made a comprehensive study of some forty amines, using copper salts as catalysts for the decomposition of the diazonium salts. His conclusions may be summarised as follows—

(a) Theoretical yields can be obtained in the cases of some mononitro- and monohalogenoanilines, except *m*-nitroaniline and analogues.

(b) In the case of aromatic amines containing no strongly electronegative substituents, replacement by the nitro group may be accompanied by the introduction of a phenolic group.

(c) Halogens exert little adverse effect on the reaction except in certain positions, when it fails.

(d) Nitro groups cannot be introduced into amines already containing three nitro groups.

(e) Better yields can often be obtained with cupric instead of cuprous salts, as also in the Sandmeyer reaction (cf. Hodgson and Sibbald²⁹).

(6) INCIDENTAL METHODS—Hantzsch and Blagden³¹ decomposed the double salt of mercuric nitrate and benzenediazonium nitrate with copper, and obtained nitrobenzene, thus showing a similar transfer of the anion from the salt to the aryl nucleus as noted in the case of halogens; e.g. Griess³² obtained chlorobenzene by the thermal decomposition of benzenediazonium platinichloride or hexachlorostannate.

Bamberger³³ decomposed benzenediazonium perbromide with caustic soda and sodium nitrite solution, and obtained a small yield of nitrobenzene.

REPLACEMENT OF THE DIAZONIUM BY THE NITRO GROUP VIA THE DECOMPOSITION OF ARYLDIAZONIUM COBALTINITRITES

Recently an entirely new development of this reaction has been introduced³⁴ by the preparation of aromatic nitro compounds via the decomposition of aryl diazonium cobaltinitrites. These complex salts are obtained as yellow to orange compounds when powdered sodium cobaltinitrite is stirred into

a well cooled neutral solution of the diazonium compound, the product crystallising out after a short interval in yields of 80–100%. Either these complexes can be isolated and then converted in a separate stage by addition to sodium nitrite solution and a copper salt, or the nitro compound can be obtained by a single-stage process involving the transitory formation of the complex followed by its immediate decomposition to the nitro compound.

Moreover, amines which cannot be diazotised by the direct method owing to diazo-oxide formation, e.g. 2-nitro-1-naphthylamine, 2:4-dinitro-1-naphthylamine, and 2:4-dibromo-1-naphthylamine, can first of all be diazotised by the nitrosylsulphuric-glacial acetic acid procedure of Hodgson and Walker⁷, and the sodium cobaltinitrite added with suitable precautions³⁵. Hot decomposition of the complex is recommended only in cases where the product has a high melting point ($> 110^{\circ}\text{C}$), since tar formation, which occurs with lower-melting compounds, decreases the yield; any diazo-oxide formed is removed by digestion with ether, in which these compounds are soluble, whereas the diazonium salts are not. The catalysts used for the decomposition of the complexes are cuprous oxide and cupro-cupri sulphite. Exceptions are *m*- and *p*-phenylenediamine, which do not behave normally.

REPLACEMENT OF THE DIAZONIUM BY THE NITRO GROUP VIA THE PREPARATION AND DECOMPOSITION OF SOLID ARYLDIAZONIUM SULPHATES

Hodgson and Mahadevan³⁶ have shown that the solid diazonium sulphates may be very serviceable for the preparation of the rarer dinitronaphthalenes, e.g. 1:2-dinitronaphthalene from 2-nitro-1-naphthylamine and 1:4-dinitronaphthalene from 4-nitro-1-naphthylamine. The solid diazonium sulphates are precipitated from the sulphuric-acetic acid diazotisation medium by ether³⁶ and washed with the same solvent. Addition of the diazonium sulphate is made to the decomposition mixture consisting of a saturated solution of sodium nitrite containing cupro-cupri sulphite in suspension³⁷. Tar formation, with consequent reduction in yield, is stated to occur if the solid diazonium sulphate is added directly to the decomposition medium, owing to overheating. There is not much difference in efficiency between this and the cobaltinitrite method. Recently, two nitro groups have been introduced simultaneously into some homonuclear diamines³⁸.

REPLACEMENT OF THE DIAZONIUM BY THE NITRO GROUP IN ALKALINE SOLUTION

A claim to have prepared nitro compounds by the decomposition of alkaline solutions of diazotised amines when boiled or steam-distilled with excess of sodium nitrite has recently been made by Hodgson and Ward³⁹, who obtained positive results from six amines, e.g. 11% of *p*-dinitrobenzene from *p*-nitroaniline.

TECHNICAL COLLEGE

HUDDERSFIELD

COLLEGE OF TECHNOLOGY

LEICESTER

(Received 20th September 1949)

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- ³⁹ Hodgson and Ward, *ibid.*, 556 (1948).

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Photochemistry in relation to Textiles

The Editor,
SIR,

Breare¹ has shown that different dyes applied to wool exert a varying protective effect against the degrading effect of light on the wool and that the protection increases with depth of shade but is not in any way related to the hue of the dye. A question at the Photochemistry Symposium² revealed that no connection had been established between the light fastness of a dye and its protective effect.

The writer makes no claim to profound knowledge of the principles of photodegradation and submits the following simple speculations in the hope that they may stimulate further thought by the specialists in this field.

Eliminating catalytic effects such as are typified by chrome treatments, it appears that light fastness of the dyes should offer an explanation of all Breare's observations.

When light energy falls on dyed wool it can be either (a) reflected, or (b) absorbed and utilised in either (i) photodegradation of the wool, or (ii) photodegradation of the dye.

No doubt all three effects occur simultaneously, and as (a) should be governed largely by depth of shade any further experimental work would require that all dyeings should be of the same intensity and not merely at equal percentages of commercial dyes. If reflection can be thus eliminated, the other two factors (b, i and ii) can be considered.

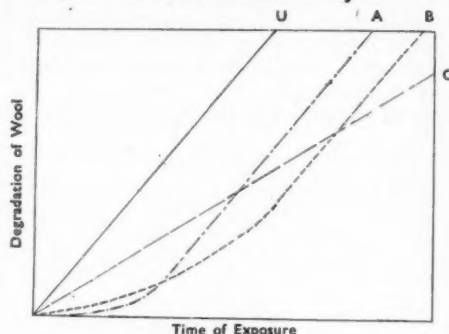
If the dye is one which is readily affected, then during the early part of an exposure a high proportion of the absorbed light energy should be utilised in degrading the dye, thus affording a high degree of protection to the wool. As the dye fades the protective effect will diminish, until the dye is completely faded and the wool unprotected.

With dyes of medium light fastness the degree of protection will be lower initially but will persist for a much longer time (both in reflection and light energy utilisation).

With a dye of maximum light fastness, reflective protection will be very persistent but light energy utilisation will be low.

From these considerations it is obvious that observations of wool degradation after a fixed period of exposure are unlikely to show any relation between protective effect and the light fastness of a dye. Thus, with a dye of poor light fastness the initial high protection followed by a period of no protection could, after a certain period of exposure, show the same degradation as that resulting from the continued partial protection of a dye of good light fastness. The protective effect must be measured, therefore, at intervals and recorded graphically.

The three types of graph (A, B, C) shown in the diagram would be produced.



- A ... Dye of low light fastness showing high initial protection which decreases at an increasing rate.
B ... Dye of medium light fastness; moderate protection slowly diminishing.
C ... Dye of maximum light fastness; constant rate of degradation, lower than that of undyed wool.
U ... Undyed wool. (For simplicity, degradation of undyed wool has been shown as a linear function.)

If these speculations are correct, they offer an explanation of Breare's observations and may also indicate the possibility of developing an analytical method of comparing light fastness of non-catalytic wool dyes based on accurate estimation of wool degradation.

Yours faithfully

W. R. LEIGH

15 ROSCOW AVENUE
BRIGHTMET
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LANCS.

13th January 1950

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- ¹ Breare, A., J.S.D.C., 65, 693 (Dec. 1949).
² *Ibid.*, p. 698.

The Editor,
SIR,

May I send a belated contribution to the discussion on P. W. Smith's symposium paper *Observations on the Anomalous Light Fastness of some Dyed Textiles*¹

There is at present little known about the actual state of a dye in the fibre after dyeing, and, until such knowledge is won, no fully satisfactory explanation can be offered for the anomalous behaviour of dyed textiles. Vat dyes and azoic dyes are, however, known to lead an independent existence in the fibre after dyeing, and individual members of the same class of dye may therefore not only be capable of interacting with each other when dyed in combination, but also with outside agencies (e.g. the crease-resist resin). Such interaction depends, of course, on the nature of the dye or, in combination dyeings, on the nature of the individual dyes.

It was, for instance, shown by the writer² that a vat dye, when dyed in combination with another, can combine with the second dye on soaping to

form a crystalline system different from those of the individual dyes. The same vat dye, however, when dyed in combination with a different partner does not behave in this way. It is evident that such a change in the state of the combined dyeing must alter the reactivity of the individual dyes, and textiles dyed with one combination will most probably behave differently towards outside agencies, e.g. light, from the textile material dyed with the other combination.

It is not suggested that this is what actually takes place in the cases mentioned by Dr. Smith, but it is put forward as a possible explanation of some of the phenomena.

The abovementioned phenomenon need not necessarily be restricted to vat or azoic dyes, but may also account for some of the other anomalies mentioned by Dr. Smith.

I should like to express my thanks to the Director of Research of the British Launderers' Research Association for permission to publish this letter.

Yours faithfully

E. KORNREICH

BRITISH LAUNDERERS' RESEARCH
ASSOCIATION

HILL VIEW GARDENS
LONDON N.W.4

20th January 1950

The Editor,

SIR,

In the course of my introduction at the Symposium¹, I expressed the hope that the discussion time would give an opportunity for the citing of cases of anomalous behaviour other than those mentioned in the paper.

In point of fact, the number of contributors on this basis was such that little actual discussion took place, but Mr. Kornreich's letter, together with Dr. Stearns's contribution to the discussion, seems to be of particular value to the dye user.

Both gentlemen stress carefully the point that it is the system as a whole which must be viewed (substrate, dyes involved, physical state of the dyes, humidity, type of light, finishing agents etc.) when assessing light fastness rather than adjudging

such a fastness to be the property of the dyes alone.

All of which leaves the dyer or printer, who must satisfy an ultimate consumer demand, very much in his present position of trying the finished product out under a given set of circumstances and assessing the result visually. More important, it causes him to think that such will always be the position, despite the undoubted contribution of the pure scientist to this very difficult field of textile chemistry.

Yours faithfully

P. W. SMITH

JAMES HARDCASTLE & CO. LTD.

BRADSHAW WORKS
NEAR BOLTON
LANCS.

13th March 1950

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- ¹ Smith, P. W., J.S.D.C., 65, 743 (Dec. 1949).
- ² Kornreich, E., *ibid.*, 62, 318 (Oct. 1946).

Colour and Fibre Thickness

The Editor,
SIR,

Various papers in the *Journal* by Boulton and Wardle, Fothergill, and Tsien and myself have drawn attention to the way in which apparent colour depends on fibre thickness. It might interest others as it interested me to learn that this was known in Roman times, two thousand years ago.

Leonard's translation of *Lucretius*, Book II, says—

Again, the more an object's rent to bits,
The more thou seest its colour fade away
Little by little till 'tis quite extinct;
As happens when the gaudy linen's picked
Shred from shred away: the purple there,
Phœnician red, most brilliant of all dyes,
Is lost asunder, ravell'd thread by thread.

Yours faithfully

J. M. PRESTON

TEXTILE CHEMISTRY DEPARTMENT
COLLEGE OF TECHNOLOGY
MANCHESTER

13th February 1950

Notes

Proceedings of the Council

At a meeting of the Council, held at the offices of the Society, 32-34 Piccadilly, Bradford, on 8th February 1950, the proceedings included the following items of interest—

A.G.M. AND DINNER 1951—It was decided that the Annual General Meeting and Dinner should be held in 1951 at Leicester on Friday, March 30th.

ANNUAL REPORT AND BALANCE SHEET—Drafts were approved for submission to the Annual General Meeting 1950.

NOMINATION OF PRESIDENT—Mr. Fred

Scholefield was formally nominated for election as President at the Annual General Meeting.

YORKSHIRE COUNCIL FOR FURTHER EDUCATION—It was reported that Mr. J. G. Evans had accepted Council's invitation to represent the Society on the Yorkshire Council for Further Education County Advisory Committee for Chemistry.

MEMBERSHIP—Twenty-four applications for ordinary, nine for junior, and four for life, membership were approved. One application for readmission was approved, and the loss by death of Mr. T. Watson was reported.

Meetings of Council and Committees March

Council—8th
Publications—14th
Colour Index Editorial Panel—20th
Textbook Subcommittee—22nd
Summer School—18th

George Douglas Lecture Committee

A small committee has been set up by Council to make arrangements for the George Douglas lectures. It will consist of the President, the President-elect or Immediate Past President, the Chairman of the Publications Committee, and a

representative of the Bradford Dyers' Association Ltd. (Mr. J. G. Evans).

The Chemical Society Research Fund

The Research Fund of the Chemical Society provides grants for the assistance of research in all branches of Chemistry, about £700 per annum being available for this purpose. Applications for grants should be submitted not later than Monday, 1st May 1950. Forms of application together with the regulations governing the award of grants may be obtained from the General Secretary, The Chemical Society, Burlington House, Piccadilly, London W.1.

OBITUARY NOTICE

Fred Townend

Born on 23rd February 1912 at Ossett, Yorkshire, Townend received his early education at the Ossett Grammar School. Matriculating in 1927, he obtained the Higher School Certificate in 1930, and he was awarded a Royal Scholarship, which enabled him to study at the Imperial College of Science and Technology in London. He gained the Associateship of the Royal College of Science and the degree



of B.Sc. (London) in 1933, and was awarded the John Perry medal for distinction in mathematics and mechanics.

Since his family was established in the Yorkshire textile industry it is not surprising that at this stage of his life Townend's interest should turn to textile chemistry, and after graduation he commenced a period of research in this field in the Textile Industries Department of the University of Leeds. The three years which he spent at Leeds were devoted to an investigation of the amino acids

of feather keratin, and for his thesis presented under this title he was awarded his Doctorate of Philosophy.

There is no doubt that the time he spent at Leeds helped to formulate his firm belief in the importance of applying pure science to industrial problems, and it was with this object in view that in 1936 he joined the staff of Imperial Chemical Industries Ltd. On their behalf his research work continued for a further eighteen months in the University of Birmingham.

When, in 1938, he moved to Manchester he commenced the investigation of problems in wool dyeing which were to be his main interest for the remainder of his life. In 1941, however, he temporarily left this field to visit the United States to obtain the benefit of American experience in the dyeing of nylon.

Although few of the results of his research work were published, he made a great contribution to the knowledge of his subject, as evidenced by his energetic participation in discussions and by his active organising ability as Honorary Secretary of the Manchester Section of the Society. He held this office for five years until April 1949, when he left Manchester to take up an appointment in Scotland.

Townend's tragic death the following July was a great loss to the Society and to the Textile Industry as a whole, for his painstaking attention to detail in his work enabled him to view the technical problems which he had to solve with an exceptional clarity. He was methodical and scrupulously honest in his approach to technology, and throughout his career his example was of great assistance, particularly to his younger colleagues.

With the same energy and interest that he gave to his work, Townend enjoyed his leisure hours. In his youth he was a keen cross-country runner and later he was fond of walking. He was an ardent bridge player and took much pride in his garden. His attitude to work and to life in general was typified by his enjoyment of the spade work which his interest in gardening entailed.

He leaves a wife and a daughter.

W. FURNESS
D. P. RAPER

New Books and Publications

Petroleum Chemicals Industry

By R. F. Goldstein. Pp. xiii + 449. London: E. & F. N. Spon Ltd. 1949. Price, 63s. 0d.

One of the most remarkable developments in chemical industry of recent years has been the rapidity with which manufacturers have turned to petroleum as a raw material. The announcement in the past few months of plans for the manufacture of petroleum chemicals in the U.K. has stimulated interest further, but has, at the same time, underlined the need for an up-to-date summary describing the separation of the raw materials and conversion of these into industrial chemicals. Dr. Goldstein's book has, therefore, appeared at an opportune moment.

From the point of view of the textile industry widespread interest already exists in petroleum chemicals, since one of the major detergents available in this country is based on higher olefins derived from a special cracking process. Non-ionic detergents are also well established here, and these are based on ethylene oxide. Other types of detergents can be based on alkylated aromatics, and manufacture of these materials will depend on separation of higher olefins and aromatic hydrocarbons. In this field Dr. Goldstein shows very clearly how petroleum can give rise to the raw materials which are required for all types of detergent.

The author traces the way in which the various types of raw material can be produced from petroleum and isolated in sufficient purity to permit manufacture of the important chemical intermediates required by an extensive range of industries. These intermediates are the simple olefins and paraffins, and the prolific American patent literature on the conversion of these intermediates to chemicals has been sifted carefully to emphasise the types of chemical rather than individual compounds which can be derived from these raw materials.

The manufacturing chemist will find, in the

chapters dealing with conversion to aldehydes, acids, esters, and olefin oxides, a wealth of invaluable information, and this section of the book is, perhaps, of more direct interest to the chemical industry than the sections which describe separation of petroleum raw materials. Nevertheless, by dwelling on the methods of the petroleum industry, the author has demonstrated a most significant factor in the manufacture of chemicals from petroleum, namely the extreme flexibility of the industry as a whole in being able to change from one type of raw material to another using the same feedstock.

Many of the chemicals to which Dr. Goldstein has referred are of great significance to the textile industry. While it is difficult to discriminate between one chemical and another, the possible manufacture of ethylene oxide from petroleum sources might be regarded as being of greatest potential benefit to the industry owing to its extreme versatility. Ethylene oxide can be converted to ethylene glycol or diethylene glycol and can be used in the manufacture of the synthetic fibre Terylene, in which the glycol reacts with terephthalic acid available from petroleum sources. Other synthetic fibres, notably nylon, are of increasing importance in the U.K., and Dr. Goldstein points out how both components of the nylon molecule may be derived from petroleum.

Undoubtedly this book is a very valuable addition to the subject, and for reference purposes it is likely to remain a standard work for some years to come. The author has utilised to the fullest degree the extensive information which exists in American patents as well as the data obtained from German chemical plant records, and the book is extremely well indexed from these aspects. In this way the author has avoided the common error of too much detail, while at the same time he gives sufficient indication to allow individual chemicals to be traced through the literature.

E. S. PAICE

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes.

Any publication abstracted may be referred to by members of the Society on application to

Dr. C. B. Stevens, Dyeing Department, Leeds University

Allied Colloids (Bradford) Ltd.

INDANTHREN GREY CL POWDER FINE FOR DYEING—This anthraquinone vat dye is produced by the Badische Anilin- & Soda-Fabrik, Ludwigshafen. It is a new product, dyeings of which on cellulosic fibres are characterised by very good fastness to light and all wet treatments including soda boiling-hypochlorite bleaching. It is very level dyeing, and can be dyed by methods IW, IK, and IN. Some fastness grades on cotton include—Light 7, chlorine 4-5, soda boiling 4.

INDANTHREN YELLOW 4GF POWDER FINE FOR DYEING—This is also a new product, giving bright, lemon yellow dyeings on cellulosic fibres. These have excellent fastness to light and wet treatments and will withstand soda boiling-hypochlorite bleaching. It is recommended for use alone or in mixtures, particularly greens with Indanthren Brilliant Green B using method IW or with Indanthren Turquoise Blue 3GK using method IK and

may also be applied by method IN in the presence of salt. Indanthren Yellow 4GF does not accelerate the tendering of cellulosic materials on exposure to light. Some fastness grades on cotton include—Light 7-8, chlorine 5, soda boiling 4-5.

FAST DYEINGS ON WOOL—This card is the French edition issued by the Société Anonyme de Matières Colorantes et Produits Chimiques Francolor describing the methods of applying their chrome, acid milling, and vat dyes to wool. It contains a range of dyeings on wool yarn of seventy-five chrome dyes, the majority of which are shown in three depths, a range of twenty-six acid milling dyes, four mordant dyes of the alizarin type, two of which are shown on both chromium and aluminium mordants, and six indigoid vat dyes on wool cloth. The optimum dyeing method and all relevant fastness data are appended alongside each set of dyeings. Finally, twenty-eight mixture dyeings in mode shades on wool yarn are also included.

DYEING OF COTTON WITH VAT DYESTUFFS—This is the English edition of the pattern book issued by Francolor describing their ranges of indigoid and anthraquinonoid vat dyes. The latter group contains fifty-seven dyes under the general title Solanthrene, which are shown in three depths on cotton yarn; while the former includes nineteen Heliane, Solane Blue, and Indigo dyes, three dyeings of which also appear. Finally, thirty dyeings on bleached cotton yarn in pastel shades and an equal number in mode shades, the majority of which have been produced by using mixtures of dyes, are included. As in the previous card, details of fastness properties and of the optimum and alternative dyeing methods are appended alongside each set of dyeings.

Clayton Aniline Co. Ltd.

CIBACET YELLOW GBA EXTRA—This dispersed dye is suitable for producing reddish yellows having good fastness to light and wet treatments on acetate rayon and nylon. Cellulosic fibres are slightly stained, but the staining can be removed by the usual permanganate-bisulphite treatment. Dyeings on nylon and acetate rayon are dischargeable to a good white, and the dye is also of interest for direct printing on these fibres if precautions are taken to avoid sublimation. Some fastness grades on acetate rayon include—Light 5, washing 5, perspiration 3-4.

FASHION SHADES—This card contains thirty-four dyeings on wool cloth to match the British Colour Council fashion colours for wool No. B 1109-1144.

FASHION SHADES FOR HALF HOSE—Fourteen dyeings on knitted wool cloth reproduce shades HH 30-44 issued by the Midland Hosiery Dyers' and Finishers' Federation and the National Hosiery Manufacturers' Federation, Leicester.

KNITWEAR—This card contains twelve dyeings on wool yarn to match B.C.C. colours No. K 188-199.

PIECE DYED SUITINGS—This card contains nine dyeings on worsted cloth matching B.C.C. colours.

WHITE DISCHARGE PRINTS—Under this title, two separate cards illustrate a range of discharge prints on cotton sateen dyed with Coprantine dyes and on viscose crepe dyed with selected Rosanthrene and Diazo dyes. The former includes fourteen prints of Coprantine dyes aftertreated with Coprantine Salt II, while the latter contains fifteen prints produced from diazotised and developed dyeings and discharged under both neutral and alkaline conditions.

CLASSIFICATION OF DIRECT COTTON DYES—This booklet contains the results obtained when the entire range of direct cotton dyes were classified according to the recommendations of the Report of the Society's Direct Cotton Dyes Committee. For convenience, the information is presented in three ways—each dye is listed, firstly in alphabetical order, secondly according to hue, and thirdly in a separate list according to whether it has been placed in Class A, B, or C.

Sandoz Products Ltd.

BRILLIANT ALIZARINE LIGHT BLUE 3F—This equalising acid dye gives pure blues considerably brighter than those obtained by using Alizarine Light Blue FF. Like the latter, it is suitable for dyeing wool in all forms, including wool and fur felt, and for printing wool and silk, and is particularly recommended for sky and baby blues where extreme brightness and fastness to light are required. Some fastness grades include—Light 5, washing at 40°C. 4, carbonising 4-5.

DERMA GREY LL—This is an acid dye for leather, particularly suitable for use on chrome upper, chrome suède, and vegetable-tanned leather for pale to medium

greys of good fastness to light, washing, acids, alkalis, and formaldehyde.

LUMIEREASE BLUE 4GL AND 2RL—These are direct dyes which give greenish and reddish blues, respectively, on cellulosic fibres. They are the first members of a series of dyes specially recommended for dyeing materials which are to be crease-resisted by the normal urea-formaldehyde method, since the light fastness of the dyeings is not adversely affected by such treatments. They exhaust well from cold dyebaths, and leave wool unstained under these conditions; they reserve acetate rayon, and the 4GL brand is completely dischargeable under both neutral and alkaline conditions. Some fastness grades include—

Blue 4GL—Light 5-6, washing 2-3, perspiration 3.

Blue 2RL—Light 6, washing 3, perspiration 3.

SOLAR BRILLIANT BLUE BRL—This direct cotton dye gives dyeings of very good fastness to light on cellulosic fibres, which become slightly greener in hue but are only slightly reduced in fastness to light when aftertreated by a normal crease-resist process. It is very level dyeing, and reserves acetate rayon completely, stains nylon slightly, and dyes silk much less deeply when applied to mixtures of these fibres and cotton. Some fastness grades on cotton include—Light 6, washing 3, perspiration 3.

VISCO MILLING RED FD (PARASULPHON SCARLET RL)—The first name of this direct dye indicates its suitability for dyeing cellulosic fibres and unions of these with wool to give dyeings of good fastness to wet treatments. Solid dyeings on cotton-wool and viscose rayon-wool materials are readily obtained. Under the title Parasulphon Scarlet RL, it is recommended for use alone or in mixtures on cellulosic materials to give dyeings fast to washing when coupled on the fibre with diazotised *p*-nitroaniline. White discharges can be readily obtained under both neutral and alkaline conditions on both the direct and aftertreated dyeings. Some fastness grades on cotton include—

Visco Milling Red RD—Light 3, washing at 105°F. 3-4, perspiration 4.

Parasulphon Scarlet RL (coupled with *p*-nitroaniline)—Light 3, washing at 105°F. 4, perspiration 4-5.

DIRECT COLOURS ON COTTON—This pattern book contains dyeings on cotton cloth in three depths of the complete range of direct dyes, including the ordinary direct, Solar, Diazamine, and Parasulphon dyes. Included alongside each set of dyeings are the usual relevant fastness data, including the effects on hue and on fastness to light and washing of the normal crease-resist process, and also the temperature of maximum exhaustion, rate of exhaustion, levelling power, and an indication of the degree of control required during dyeing for producing satisfactory dyeings.

NYLON HOSIERY COLOURS—This pattern book contains seventeen dyeings on woven nylon of dyes selected from the dispersed, direct, chrome, acid milling, and equalising acid classes and twenty-four dyeings on knitted fabric produced with either one or more of the first series to give a variety of browns, fawns, and greys. Two patterns of each dyeing are so mounted that they are seen against a flesh-coloured background, and in one case the appearance during wear of a single thickness of material can be estimated, while in the other the colour of four layers of the single-thickness dyeing can be assessed. With the deeper dyeings, especially the blacks, the differences are most marked.

FASHION SHADES ON WOOL-NYLON GABARDINE—This card contains forty dyeings on cloth woven from wool-nylon mixture yarns. The majority have been produced using Xylene Fast P dyes, Resoline B being added as an assistant for producing solid dyeings in the case of the paler shades.

Reports on German Industry

Steinkohlenbergwerk Rheinpreussen, Homberg

Wetting Agents and Detergents from

Fischer-Tropsch Products

FDX 713* (PB 82,011; Microfilm X 117).

Among a number of reports, written in German during

1938-46, there are some which deal with the production of soap from synthetic fatty acids and two, dated 1942, on practical tests made with detergent, emulsifying, and wetting agents made from Fischer-Tropsch products.

C. O. C.

* Material available for inspection at, and photocopies available from, T.I.D.U., Board of Trade, 40 Cadogan Square, London S.W.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.

I.G. Farbenindustrie A.-G., Leverkusen**Detergents and Auxiliary Agents—Dyes Intermediates for Dyes and Auxiliary Agents**

FDX 681* (PB 73,896; Microfilm N 58).
The first part of this microfilm consists of 16 reports, dated 1940–1944, on various finishing agents, mainly water-repellent agents. The second part consists of 37 reports, dated 1929–1940, on research work on dyes. The third part contains 32 reports, dated 1929–1936, on work on intermediates for dyes and auxiliary products and a report on mothproofing agents made from organometallic compounds, lists of the various compounds tested and the results being given. C. O. C.

I.G. Farbenindustrie A.-G., Leverkusen**Mothproofing Agents—Dyes and Intermediates**

FDX 683* (PB 73,908; Microfilm N 60).
Microfilm of 14 reports, in German, on the testing of 216 derivatives of urea and thiourea and similar compounds, phenyl salicylate and nine related compounds, Meerwein's alkoxides, acetylated sugar and derivatives of saccharin, quinoline and plasmoquin, metal compounds of 1:3-diketones, condensation products of isatins with substituted phenols, 95 bases containing at least one aliphatic chain and 75 benzene-soluble compounds, for mothproofing properties. There are also 32 reports on research and testing work on dyes and intermediates. C. O. C.

I.G. Farbenindustrie A.-G., Leverkusen**Periodical Research Reports 1941–43 New Compounds and their Application in the Manufacture of Textiles, Leather, Dyes, Paints, etc.**

FDX 701* (PB 74,639; Microfilm L 90).
A large number of reports (850 pp.), written (in German) during 1941–43, dealing with the preparation and use of new compounds including many new dyes, water-repellent materials, many isocyanates, resins, mothproofing agents, etc. C. O. C.

I.G. Farbenindustrie A.-G., Ludwigshafen**Comprehensive Report on the Dyeing of Textile Fibres—Water Treatment—Hydrosulphite—Titanium Dioxide—Copper Phthalocyanine**

FDX 684* (PB 73,758; Microfilm X 5).
A microfilm of a number of reports, written in German in 1936–37. One (184 pp.) is a thorough review of textile dyeing divided into the following sections—(a) fibres and fabrics, (b) acid dyes, (c) direct dyes, (d) Naphtol AS dyes, (e) Alizarin, (f) Aniline Black, (g) Noir réduit, (h) sulphur dyes, (i) vat (including Indigosol) dyes, (k) basic dyes, (l) acetate rayon dyes, (m) theory of dyeing, and (n) bibliography up to 1935. There are three reports on the manufacture of hydrosulphite and several on water conditioning. The various processes for making the various types of Heliogen Blue B and of Heliogen Blue NCB and Vulcan Fast Blue BF are discussed in a report written in 1937. A detailed report (100 pp.), written by B. Wurzschnitt in 1938, deals with all aspects of the use of titanium dioxide as a white pigment from both theoretical and practical points of view. It is considered not to be chemically inert but a surface-active pigment having considerable effect on the system in which it is incorporated, and a new grouping of pigments based on this fact is proposed. C. O. C.

I.G. Farbenindustrie A.-G., Ludwigshafen**Aluminium Oxalate Complexes as Substitutes for Djirak in Batik Dyeing**

FD 1511/49* (PB 70,192).

Describes *inter alia* the production and testing of a number of aluminium oxalate complexes, which are far superior to the natural product Djirak in Batik dyeing. C. O. C.

I.G. Farbenindustrie A.-G., Hoechst**Flameproofing Agents for Textiles—Luminous Paints—Tanigans**

FDX 700* (PB 70,155; Microfilm L 1).

Among numerous miscellaneous reports there are some correspondence and notes (50 pp.), dated Jan.–Aug. 1941, on sulphurylamide and other flameproofing agents. There are a considerable number of reports (112 pp.), dated 1939–43, on various aspects of luminous pigment and paint production, including details about the manufacture of various types of Lumogen E, L, Lg, and Z and Luminant Blue and Yellow. The production is described of many of the Tanigan brands. C. O. C.

I.G. Farbenindustrie A.-G.**Sulphonic Acids of Pyrene and their Derivatives**

E. Tietze and O. Bayer. FD 2045/48.

Photocopy of a 22-pp. article, in German. Exhaustive research work carried out in 1933–39 on the sulphonic acids of pyrene and their derivatives and the production from them of components for azo dyes is described in great detail, many data on compounds being given together with their structural formulae. C. O. C.

I.G. Farbenindustrie A.-G., Leverkusen**Noiseless Condensation of Steam Determining the Properties of Textile Fibres and their Suitability for Special Fabrics Triaceta Fibre**

FDX 674* (PB 73,567; Microfilm T 6).

Among a large number of research reports, in German, is one on the reduction of the noise caused when water is heated by direct steam; another is in two sections, the first of which deals with the qualitative valuation of fibres and the second with methods of determining the suitability of fibres for making up into special yarns and fabrics. A third report is a comprehensive review of the work carried out in developing Triaceta fibre and its dyeing and finishing. C. O. C.

I.G. Farbenindustrie A.-G., Ludwigshafen**Methods of Analysis and Test**

FDX 680* (PB 73,503; Microfilm G 28).

A collection of numerous analytical and testing methods (in German) for inorganic and organic compounds, tanning agents, food dyes, etc. C. O. C.

Survey of the Use of Infra-red Spectra in Chemical Analysis in Germany

FD 1054/49* (FIAT 708; PB 48,678).

A report, in English (76 pp.), by an Allied investigator, together with copies of notes, papers, etc. in German (143 pp.), on the use of absorption spectroscopy, which has not been so general for analysis in Germany as in the U.S.A. Infra-red spectra were used for analysis only in three or four places, while ultra-violet absorption spectra were used fairly generally for identifying compounds and for qualitative analysis. Raman spectra were used for analysis of mixtures, for identification of compounds, and for determining molecular structures. The design and operation of the apparatus used are discussed. C. O. C.

* Material available for inspection at, and photocopies available from, T.I.D.U., Board of Trade, 40 Cadogan Square, London S.W.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.

Abstracts from British and Foreign Journals and Patents

(The Titles of Patents are abridged and modified)

I—PLANT; MACHINERY; BUILDINGS

Cutting Down Steam Costs. C. T. Wade. *Dyer*, 102, 377–381 (7th Oct. 1949).

Some of the main causes of heat losses in the various dyeing and finishing processes are indicated, and it is shown how in some cases the use of low-grade heat is just

as effective as high-grade heat. The design of plant to reduce the overall fuel consumption is considered. H. K.

Automatic Temperature Control. L. Walter. *Dyer*, 101, 36–46, 156–167, 270–279 (14th Jan., 11th Feb., 11th March 1949).

Improved dyeings and economy in fuel and steam can

be attained by automatic temperature control. It is shown that hand control of steam valves is never entirely satisfactory even with an efficient operator. Control can be achieved by (a) throttle control of the steam valve as the temperature rises; (b) shutting off steam when the desired temperature has been reached and then admitting more steam as the temperature falls below a certain value; (c) the "elapsed time" method, where throttle control is employed but the process is finished automatically after a certain time has elapsed at a predetermined temperature; (d) "programme control", where the temperature can be raised at a desired rate, kept steady for the required time, and lowered gradually, the process being finished completely. The characteristics of these various controllers are described along with methods of installation for improving existing equipment. Each type of instrument has its definite field of application, and the fitting of the controller and relative position of the thermostat on various types of dyeing machine are described. Some practical examples of methods of minimising heat and steam losses from dye vessels are given. H. K.

PATENTS

Apparatus for Intimately Contacting Two Immiscible or only Partly Miscible Liquids. N. V. De Bataafsche Petroleum Maatschappij, R. B. Olney, R. L. Maycock, and G. M. Hartwig. B.P. 632,888.

Pelleting of Carbon Black. Columbian Carbon Co. and L. J. Venuto. B.P. 633,019.

Pelleting of Carbon Black. R. W. Skoog and United Carbon Co. Inc. U.S.P. 2,480,782.

Processing Yarn. A. L. Ewing and Du Pont. U.S.P. 2,481,916.

The yarn passes around a yarn-advancing reel which rotates closely enough above a shallow-bottom dip pan for the yarn on the lower half of the reel to pass through the liquid in the pan. C. O. C.

Drying Coated Thread. D. K. Alpern, H. M. Freyberg, W. H. Hawkins, and Freyberg Bros.—Strauss Inc. U.S.P. 2,483,378.

Cloth Feed Control. F. T. Miller and United Merchants & Manufacturers Inc. U.S.P. 2,482,497.

In the continuous wet processing of cloth where it is passed from one driven roller to another driven roller, putting a J-box for the cloth between the two rollers prevents any tension from being applied to the cloth by any alteration in the speed of either or both rollers. The J-box is pivoted horizontally, there being stops limiting its motion in either direction. The box is so biased relatively to its pivot axis as to move to engage one of the stops when the box becomes full and the other stop when the box is only partly full. A circuit control for the motor driving the second of the rollers is responsive to movements of the box. C. O. C.

Continuous Open-width Dyeing Machine. J. Dugler. B.P. 632,975.

The cloth is passed without tension through the machine, where the liquor is passed through the cloth by forced or induced circulation. C. O. C.

Pile Fabric Dyeing Machine. K. Dutton, J. E. Holt, R. H. Sennett, and I.C.I. Ltd. B.P. 633,046.

Modification of B.P. 609,411 (cf. J.S.D.C., 65, 75 (Feb. 1949)). Sideways flow of dye liquor can be avoided and uniform impregnation ensured by using instead of the two flanged rollers any device which forms a slot of dimensions equal or nearly equal to the width and depth of the pile fabric. Such a device can be a simple slot in a sheet of metal or preferably two rollers of uniform thickness, the ends of the slot being formed by separate pieces of metal or other suitable material. C. O. C.

Printing Bed for Screen Printing Machines. L. Martin. U.S.P. 2,480,493.

A pair of parallel endless bands are supported by pulleys at their looped ends. Slat is fastened across these bands at regular intervals with their ends in alignment. A continuous blanket is secured to the slats. A flat table supports the upper reach of slats. Guides extending along the table prevent lateral movement of the slats. The drive for the bands is co-axial with the pulleys at one end of the bands. This construction ensures accurate registration when several screens are used in succession; in particular

any side or end play of the fabric being printed is prevented. C. O. C.

Tenter specially suitable for Lace. J. A. Grundy and John Bromley & Sons Inc. U.S.P. 2,482,270.

A tenter is described which has automatic control and feed and which may form a single unit together with the loom and the "dunker" (sizing machine). C. O. C.

Fabric-folding Machine. G. Haerberlin and Richmond Pierce Dye Works Inc. U.S.P. 2,482,764.

A machine for the zigzag folding of pieces. C. O. C.

Ultra-violet Radiation Treatment of Textiles after Laundering or Dry Cleaning. L. Silver and V. A. Douris. B.P. 631,532.

Modification of B.P. 592,707 (cf. J.S.D.C., 64, 127 (March 1948)), the means for charging and discharging the machine being simplified. C. O. C.

Forming Hat Bodies from Fibrous Webs. Merrimac Hat Corp. B.P. 632,310.

II—WATER AND EFFLUENTS

Problems arising from the Disposal of Effluents containing Synthetic Detergents. H. H. Goldthorpe, W. H. Hillier, C. Lumb, and A. S. C. Lawrence. *Chem. and Ind.*, 679-682 (1st Oct. 1949).

Sewage purification requires the removal of settleable solids from polluted water, and unloading devices have been based on the properties of soap as the traditional detergent. With synthetic detergents the protective layer surrounding the fine particles is not so readily destroyed, and the preliminary unloading process of sedimentation is less efficient. In the wool textile districts this loss of primary sludge and the increased loading at the secondary biological purification stage are regarded with alarm. The possible effects of synthetic detergents on the plant and animal life employed in the secondary stage are also causing apprehension. Additional contributions are made in a discussion on the paper. G. L.

PATENT

Preventing Foaming of Water. I.C.I. Ltd. B.P. 632,242.

Ricinoleoylamines (obtained by heating ricinoleic acid containing inter- and intra-esters with water and an amine at $> 100^{\circ}\text{C}$. and $> 60\text{ lb./sq.in.}$ until the esters are hydrolysed, releasing the pressure and allowing the steam to escape, and finally heating the resultant mixture at $> 150^{\circ}\text{C}$. without causing dehydration of the acid) are excellent antifoaming agents for use in boiler waters. C. O. C.

Flax Wet Spinning Frame as a Source of Catalytic Damage in Bleaching. W. Honneyman. (XIV, p. 255.)

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Emulsifying Power of Detergents. J. P. Sisley, Reutenauer, and Sicard. *Teintex*, 14, 361-369 (Sept. 1949).

A new method for measuring the emulsifying power of detergents has been devised. It is an adaptation of the experimental method used in an American standard "Steam Emulsion Test" (A.S.T.M. D-137.36). A current of steam is blown for 7 min. through a 10-c.c. portion of arachis oil and detergent contained in a test-tube. The test-tube is then allowed to stand for 10 min., when some separation into an upper layer of emulsion and a lower layer of water takes place. The emulsifying power of the detergent is the percentage of water contained in the upper layer after this time. Full experimental details are given, and results are quoted for a series of soaps and synthetic detergents. F. A.

Modern Surface-active Agents. A. A. Ermolaeva. *Tekestil. Prom.*, 9, 25-28 (April 1949).

An examination is made of a number of surface-active agents, mainly of German and American manufacture. Tables are given of their capillary, wetting, emulsifying, detergent, and other properties. Their behaviour as kier-boiling and bleaching assistants is also examined. A. E. S.

Effect of Temperatures on Finishing Compounds.

A. H. Noble. *Amer. Dyestuff Rep.*, 38, P 115-P 121 (7th Feb. 1949).

Sulphated oils, sulphated tallows and fatty acid softeners, synthetic substantive softeners, and weighting substances were subjected to scorch, rancidity, and accelerated ageing tests. A comprehensive survey of the results is given together with indications of the suitability or otherwise of the products tested. H. K.

PATENTS**Viscous Water-soluble Cellulose Ether Solutions.**

C. R. W. Morison and Carbide & Carbon Chemicals Corp'n. U.S.P. 2,480,949.

The viscosity of aqueous solutions of cellulose ethers can be largely increased by adding a small amount of a water-soluble alginate. C. O. C.

Oxycellulose Products suitable as Sizes for Paper, Textiles, etc.

W. H. Van Delden, J. B. Rust, and Montclair Research Corp'n. U.S.P. 2,482,042.

Cellulose, degraded with an aqueous solution of a base in presence of oxidising agents until the product has as a 1% solution in 8% aqueous sodium hydroxide a viscosity of 1.9-4.0 centipoises at 28.5°C., when dissolved in dilute alkalis yields solutions of satisfactory consistency and viscosity for use as sizes which form water-insoluble, continuous coherent films upon coagulation. C. O. C.

Water-insoluble Starch Finish for Textiles.

N. V. W. A. Scholten's Chemische Fabrieken. B.P. 632,786.

A wash-resistant finish is imparted by impregnation at 40-50°C. with a solution containing starch or a starch derivative, an aminotriazine containing > 1 reactive H atom, and an aldehyde capable of forming a synthetic resin with the triazine. Instead of the triazine and the aldehyde the solution may contain their precondensate, which, if desired, may be etherified and/or esterified. The solution contains enough of a non-volatile or only slightly volatile acid and/or of a substance yielding acid when heated to give the solution pH 5 at 40-50°C., maintain it at that pH for not < 2 hr., and act as an acid catalyst in the final baking. C. O. C.

Soaps which do not form Coagulable Lime Soaps.

Lever Brothers & Unilever Ltd. B.P. 631,421.

Addition to soap of a little of a salt of a higher ($C_{18}-C_{21}$) fatty acid ester of an *N*-(hydroxyalkyl)amide of a lower ($< C_7$) sulphocarboxylic acid, e.g.—



results in any lime soaps formed when the soap is used in hard water being kept in a finely dispersed state. C. O. C.

Preventing Deposition of Lime Soaps.

H. H. Young, D. Rubinstein, and Swift & Co. U.S.P. 2,483,253.

Addition to a soap of 5-50% of its weight of an acylated bis(hydroxyalkyl)amine prevents deposition of insoluble metal soaps even during rinsing when the soap is used in water containing Ca, Mg, or other multivalent metal salts. C. O. C.

Emulsifying Agent.

Co-operative Wholesale Society Ltd., L. O'Brien, and A. Glover. B.P. 632,149.

Stable aqueous emulsions of oils and waxes are obtained by using as the emulsifying agent a mixture of a glyceryl stearate, a lipid, e.g. a higher fatty alcohol, and a free fatty acid which is solid at normal temperatures. C. O. C.

Emulsifying, Detergent, and Wetting Agents.

American Cyanamid Co. and E. A. Vitalisi. B.P. 632,321.

A mixture of 100 parts by weight of an alkali-metal or ammonium salt of a dialkyl sulphosuccinate whose alkyl groups contain 5-10 C and 5-100 parts of an alkali-metal or ammonium benzoate is readily soluble in water and remains as a free-flowing powder even after long storing. C. O. C.

Polyacrylamides — Protective Colloids.

General Aniline & Film Corp'n. and G. D. Jones. B.P. 631,738.

Products having excellent protective colloid action are obtained by treating an aliphatic polyaminocarboxylic acid, in which the amino groups are primary or secondary, with acrylic or an α -substituted β -unsubstituted acrylic acid halide or anhydride, the molar amount of halide or anhydride being less than the number of amino groups in the polyamino acid, e.g. *N*-methacryloyllysine. W. G. C.

Organic Phosphites—Flameproofing Agents.

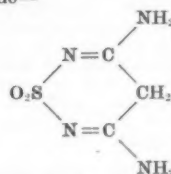
J. Truhlar, A. A. Pantisios, and R. F. Flavyty. U.S.P. 2,480,790.

Organic phosphites are more effective flameproofing agents and much less soluble than the corresponding phosphates. They are good plasticisers and solvents for synthetic polymers. Addition of 10-20 parts by weight of an organic phosphite to 100 parts of a mixture of chlorinated wax and chlorinated naphthalene completely eliminates any tendency for material treated with the mixture to be damaged by liberation of HCl. C. O. C.

Resins from 3:5-Diamino-4-dihydrothiadiazine-1-dioxide for treating Textiles, Stoving Lacquers, etc.

H. A. Walter and Monsanto Chemical Co. U.S.P. 2,479,441.

The resins obtained by treating 3:5-diamino-4-dihydrothiadiazine-1-dioxide—

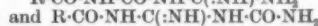
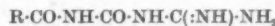


with aldehydes can be used as the basis for moulding powders which yield products of exceptional toughness and resilience. Etherification of these resins, preferably by forming them in presence of an alcohol, yields light-coloured liquids, soluble in alcohol and aromatic hydrocarbons, which give hard tough films on curing at 100-200°C. As these ether resins cure rapidly at relatively low temperatures in absence of an acid catalyst, they are especially suitable for the treatment of textiles. C. O. C.

Amino Resin Accelerators.

British Industrial Plastics Ltd. and D. E. Nagy. B.P. 632,335.

Salts of acylcarbamylguanidines or of acylamidinouras, probably—



respectively, e.g. neutral acetylcarbamylguanidine sulphate, are accelerators for amino resins; they effect rapid curing at 120°C. but do not cause premature curing on storage. E. C.

Concentrated Dispersions of Polytetrafluoroethylene.

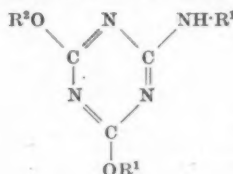
Du Pont. B.P. 631,504.

Concentrated aqueous dispersions of polytetrafluoroethylene are prepared by adding a dispersing agent to a dilute aqueous dispersion (as produced by emulsion polymerisation), coagulating the dispersion, decanting the clear supernatant liquid, and redispersing the coagulated polymer in its retained water. E. C.

Resinous Compositions for treating Textiles and Leather.

F. C. Schaefer and American Cyanamid Co. U.S.P. 2,481,155.

The resinous materials obtained by treating an aldehyde with the thermoplastic composition obtained by a transesterification reaction between a triazine of formula—



($R^1 = H$ or a univalent hydrocarbon radical, $R^2 =$ a univalent hydrocarbon radical) and a dihydric alcohol (containing no tertiary hydroxyl group, an O atom of a linear ether in a hydrocarbon chain, and no epoxy group) are useful as finishes for textiles and leather, laminating textiles or paper, coating compositions, etc. U.S.P. 2,481,155.

The thermoplastic composition is replaced by a polyhydric alcohol containing only primary and secondary hydroxyl groups and no other functional group. C. O. C.

Stable Aqueous Emulsions of Drying or Semi-drying Oils. Société l'Impregnation S.A.R.L. B.P. 631,527.

The oil is added in a thin stream to an aqueous liquid at 50–60°C. containing an emulsifying agent, with simultaneous agitation of the liquid. C. O. C.

Modified Phenolic Resin. Quaker Oats Co.

B.P. 631,109.

An alkali-catalysed stage II phenolic resin is heated at < 100°C. with a monomeric or polymeric furfuryl alcohol to produce a thermosetting resin, suitable for bonding laminates, which has better resistance to alkalis when cured than unmodified phenolic resins. E. C.

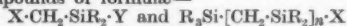
Laminating Resin. N. M. Boyts, J. M. J. M. Estevez, W. R. Davis, and I.C.I. Ltd. B.P. 632,177.

A liquid composition suitable for impregnating or low-pressure laminating fabrics, which is polymerised with a peroxide catalyst at 100°C., consists of 1–50% of a plasticiser, e.g. dioctyl phthalate or tritolyl phosphate, 2–40% of a liquid containing one vinyl or vinylidene group per mol., e.g. butyl methacrylate, and > 20% of one or more compounds containing at least two such groups separated by an average of 8–45 atoms, of which > 2 are O and/or N, e.g. the dimethacrylates of a low-mol.wt. dihydroxyethylene glycol phthalate. E. C.

Organosilicon Polymers—Waterproofing Agents. Dow Corning Corp. and J. T. Goodwin.

B.P. 631,619.

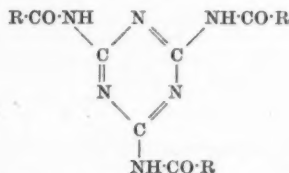
Organosilicon polymers of formula $R_2Si[CH_2SiR_2]_mY$ are prepared by treating with an alkali metal a mixture of two compounds of formulae—



(R = Alk or C_6H_5 , X = Hal, Y = Hal or O-Alk, n = 0 or an integer, m = an integer). The products are useful as waterproofing agents. E. C.

Triacetylmelamines—Water-repellent agents. Monsanto Chemical Co. B.P. 631,757.

Triacetylmelamines of formula—



(R = C_6H_5 , alkyl or alkenyl) are prepared by heating mixtures of melamine and a C_{10} – C_{30} saturated or ethylenically unsaturated acid anhydride. W. G. C.

Rot- and Insect-proofing Agents for Textiles, Furs, Leather, Paper, etc. B. F. Goodrich Co., W. D. Stewart, and J. H. Standen. B.P. 632,154.

Dispersions of the addition product of the zinc salt of an N-substituted dithiocarbamic acid with an amine having at least 1 H in an amino group are used. C. O. C.

Rotproofing and/or Mothproofing Agents. Ward Blenkinsop & Co. Ltd. and G. J. Pritchard.

B.P. 632,710.

The products obtained by treating compounds of formula A-R-X-R-A (A = SO_2H , COOH, or OH; R = a subst. or unsubst. mono- or polynuclear arylene group; X = O, S, S-S, SO_2O , or, where A = COOH, CH_2 or $CH(CH_3)$) with salt or salt-complex forming organo-metallic compounds having rot- and/or mothproofing properties are used. They have better fixation to the material to be treated than if only the second component is used. C. O. C.

Insecticidal Compounds suitable for Mothproofing. Geigy Colour Co. Ltd. and I. E. Balaban.

B.P. 632,635.

Compounds of formula $R^1CCIR^2CCl_3$, obtained by chlorinating a compound of formula $R^1CHR^2CCl_3$ or $R^1CR^2CCl_3$ (R^1 and R^2 = phenyl which may be substituted by Alk, Alk-O, or Cl), are used for rendering textiles, paper, etc. unsuitable as either food or habitation for insects. C. O. C.

Vinyl Resin Stabilisers. U.S. Rubber Co.

B.P. 631,909.

Polyvinyl chloride and its copolymers are stabilised to light by incorporating 0.5–3% of an alkali-metal pentavalent triphosphosphate of formula $M_2R_5(P_2O_5)_2$ (R = octyl or 2-ethylhexyl, and M = alkali metal). W. G. C.

Highly Flexible Wrinkle Coatings. New Wrinkle Inc.

B.P. 632,787.

An agent which when added to wrinkle coating compositions imparts high flexibility to the resulting coatings comprises wrinkling oil (10–20 gal. (U.S.A.)), non-drying oil (10–20 gal. (U.S.A.)), and wrinkle-producing drier (0.5–1.5 lb.). The wrinkling oil is heated at 530–540°F. with some of the non-drying oil until gelation is about to start, and the remainder of the non-drying oil added. The mixture is heated to 450°F. when the drier is added, and the whole heated at 500°F. until homogeneous. C. O. C.

Odourless Phenol-Formaldehyde Resins for Impregnating Cellulosic Textiles and other Fibrous Products. J. J. Wachter and Westinghouse Electric Corp. U.S.P. 2,482,525.**Dicyandiamide-Aniline Condensates—Dye Intermediates and Agents for Preventing Gas Fading of Dyed Cellulose Acetate.** D. W. Jayne, Jr., H. M. Day, and American Cyanamid Co. (IV, p. 242.)**Salts of Phenol-Formaldehyde Condensates.** C. J. Pedersen. (XI, p. 251.)**Methylolmelamine Ether Resins modified with Orthoesters of Silicon.** S. H. Rider and Monsanto Chemical Co. (XIII, p. 252.)**IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS****Behaviour of Nitrophenols with p-Toluenesulphonyl Chloride. V—Abnormal Behaviour of some Dinitrophenols with a Methyl Group in the meta-Position.** S. S. Joshi and B. B. L. Kapoor. *J. Indian Chem. Soc.*, 26, 539–540 (Nov. 1949).

4-Bromo-3-methyl-2:6-dinitrophenol, 3:5-dimethyl-2:4-dinitrophenol, and 4-chloro-3:5-dimethyl-2:6-dinitrophenol all form p-toluenesulphonic esters in sodium carbonate solution. The first, however, forms only the sulphonate when heated with the acid chloride and diethylaniline, while the latter two do not form even an ester under these conditions. In none of these cases is the hydroxyl group replaced by chlorine. The esters are quite stable and non-reactive towards ammonia and aniline. H. H. H.

Studies of Nitro-m-Cresols and their Halogen Derivatives. I—6-Bromo-2:4-dinitro- and 4-Bromo-2:6-dinitro-m-cresols. S. S. Joshi and S. P. Gupta. *J. Indian Chem. Soc.*, 26, 541–542 (Nov. 1949).

Bromination and subsequent nitration of m-cresol affords 6-bromo-2:4-dinitro-m-cresol, m.p. 106°C. (previous literature, m.p. 115°C.), and 4-bromo-2:6-dinitro-m-cresol, m.p. 78°C. The latter has been prepared also by bromination of 6-nitro-m-cresol and subsequent nitration of the 4-bromo-6-nitro-m-cresol thus obtained. H. H. H.

2-Chloro-5-nitroaniline. C. Buchanan and S. H. Graham. *J.C.S.*, 3435–3436 (Dec. 1949).

The following procedure proved to be more satisfactory for the preparation of 2-chloro-5-nitroaniline than nitration of o-chloroaniline sulphate (Chattaway,orton, and Evans, *Ber.*, 33, 3062 (1900)) or reduction of 1-chloro-2:4-dinitrobenzene (Claus and Stiebel, *Ber.*, 20, 1379 (1887)), viz. N-o-chlorophenylphthalimide was nitrated to N-(2-chloro-5-nitrophenyl)phthalimide, which hydrolysed with 2N-NaOH to the above amine (cf. also Brady, *J.C.S.*, 127, 2264 (1925)). H. H. H.

Monochlorination of 3-Fluoro-6-nitrophenol in the 4-Position. H. H. Hodgson and J. Nixon. *J.C.S.*, 3437 (Dec. 1949).

When 3-fluoro-6-nitrophenol is stirred at room temperature into 1.45 N. sodium hypochlorite, only monochlorination occurs, to give 4-chloro-5-fluoro-2-nitrophenol. The latter compound was synthesised from 4-chloro-3-fluoroanisole by demethylation followed by nitration of the 4-chloro-3-fluorophenol with nitrous acid. H. H. H.

Some Diazoamino- and Aminoazo-naphthalenes.

H. H. Hodgson and J. Habeshaw. *J.C.S.*, 3436-3437 (Dec. 1949).

To obtain further evidence of isomeric changes of the diazoamino-aminoazo type in the naphthalene series, 2-nitro-, 4-nitro-, and 4-chloro-1-naphthylamine were treated in ethanol solution or suspension with anil nitrite in the presence of less H_2SO_4 or HCl than was necessary for the formation of the diazonium sulphate or chloride. The 4-substituted compounds gave their respective aminoazonaphthalenes via their unstable diazo-amino isomerides, but 2-nitro-1-naphthylamine gave 2:3'-dinitro-4'-amino-1:1'-azonaphthalene directly, no intermediate diazoamino compound being observed. These reactions indicate that coupling in the *ortho* position to an amino group in the naphthalene series may be preceded by formation of the isomeric diazoamino compound.

H. H. H.

Demethylation of NN-Dimethyl-p-Aminoazobenzene (Butter Yellow) with Hydrogen Peroxide.

W. Anderson. *Nature*, 163, 444-445 (19th March 1949).

The products obtained when Butter Yellow is treated in *tert*-butanol with hydrogen peroxide in presence of an osmium tetroxide catalyst are being studied. So far *N*-methyl-*p*-aminoazobenzene, *p*-aminoazobenzene, and *p*-nitroazobenzene have been isolated.

A. S. F.

Effects of Sodium Chloride, Pyridine, and Ethyl Alcohol on the Absorption Spectra of certain Azo Dyes in Aqueous Solution.

J. T. Martin and H. A. Standing. *J. Textile Inst.*, 40, T 671-T 688 (Oct. 1949).

The effect of sodium chloride on the absorption spectra of Benzopurpurine 4B (*Colour Index*, No. 448), its *meta* isomer, Direct Fast Orange SE (*C.I.* 326), Chrysophenine G (*C.I.* 365), and Naphthalene Orange G (*C.I.* 151) is related to the capacity of the dye to aggregate in aqueous salt solution. The spectrum of each dye in aqueous pyridine or aqueous ethanol depends on the concentration of pyridine or ethanol, in absence of salt. This is attributed mainly to the formation of a complex with the dye in the case of pyridine, and to changes in dielectric constant when ethanol is added. When dissolved in 10% aqueous pyridine or in 50% aqueous ethanol, none of the dyes shows any marked change in its absorption spectrum on addition of salt.

A. S. F.

Effects of a Polyethylene Oxide Condensate and some Polyethylene Glycols on the Absorption Spectra of certain Azo Dyes.

J. T. Martin and H. A. Standing. *J. Textile Inst.*, 40, T 689-T 701 (Oct. 1949).

Dispersol A, a polyethylene oxide condensate, has an effect similar to that of pyridine on the spectrum of Benzopurpurine 4B (*C.I.* 448). This effect is independent of Dispersol A concentration over the range 0.5-10%. The presence of 1% of a polyethylene glycol has an effect similar to that of Dispersol A, and the effect is greater as the mean mol. wt. of the glycol increases. Estimation of the relative effects of Dispersol A and a polyethylene glycol indicates that, although complexes with the dye may be formed in both cases, the dye-glycol complex is less stable than the dye-condensate complex, possibly owing to the presence of micelles in the condensate.

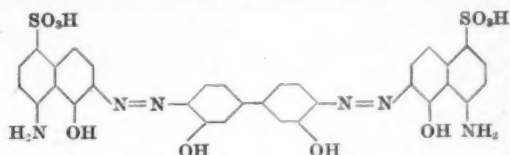
A. S. F.

Copper-containing Dyeings and Dyes.

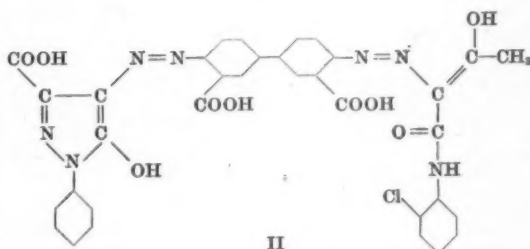
E. Krähenbühl. *Textil-Rund.*, 4, 157-168 (May 1949).

The formation of co-ordination compounds between metals and dyes is discussed first generally and then in detail for the combination of copper with direct dyes. Many of the earlier direct dyes whose fastness properties are improved by aftercoppering contain an *o*-hydroxyazo group. Examples are Diamine Pure Blue FF, Diamine Blue RW, and Chicago Blue B, which are all prepared by coupling dianisidine with naphthols. The light fastness of these dyes is greatly improved by aftercoppering, whilst their fastness to washing is slightly improved. Other dyes, which do not contain an *o*-hydroxyazo group, combine with copper, but the copper is not fast to washing. It is probable that part of the sodium in the sodium sulphonate groups is replaced by copper. Precoppered direct dyes have been prepared which are analogous to the prechromed dyes used for wool, e.g. by coupling diazotised *o*-aminophenol with a derivative of *J*-acid so as to obtain

two hydroxy groups adjacent to the azo group. The copper complexes of these dyes, unlike those of dyes based on dianisidine, are sufficiently soluble to be applied in the usual manner. Examples of this type of dye are Chlorantine Fast Violet 2RLL, Chlorantine Fast Brown 8RLL, and Chlorantine Fast Violet 5BLL; the light fastness properties of these dyes are illustrated by colour plates. A further advance was the preparation of dyes which form copper complexes of increased fastness to both light and washing. Two such dyes (I and II) are of the benzidine



I



II

type like the Diamine Pure Blue group but with the methoxyl groups of the dianisidine replaced by hydroxyl or carboxyl groups. The carboxyls act as solubilising groups in place of the usual sulpho groups, but lose their solubilising action when involved in complex formation with copper. When direct dyes are aftertreated with preparations containing both copper salts and cation-active agents, the water and washing fastnesses may be improved but the light fastness may or may not be improved, because the copper salt and the cation-active agent have opposing influences on this property. When such an aftertreatment is applied a judicious selection of dyes is therefore required. Many Coprantine dyes are suitable. Instead of being applied with Coprantine Salt II in one bath they are applied like ordinary direct dyes and then aftertreated with the copper preparation in a fresh bath. This is an advantage when dyeing dark shades in a standing bath. The application of the process to printing and continuous dyeing is also described. In a discussion of this paper R. Wizinger (*ibid.*, 4, 216-219 (June 1949)) emphasises the part played by ring formation in metal-dye complexes.

F. A.

Determination of the Constitution of Anthraquinonoid Vat Dyes.

W. Wittenberger. *Melliand Textilber.*, 30, 159-160 (April 1949).

Recommendations are given for the procedure to be followed in determining the chemical constitution of anthraquinonoid vat dyes. The methods whereby the constitutions of Indanthren Yellow 4GK, Bordeaux B, and Olive T were determined are given as illustrations. F. A.

5:5'- and 7:7'-Difluoroindigo.

A. Roe and C. E. Teague. *J. Amer. Chem. Soc.*, 71, 4019, 4020 (Dec. 1949).

5:5'- and 7:7'-Difluoroindigo have been synthesised in several ways; they vat with hydrosulphite as easily as does 5:5'-dibromoindigo and the stability of the three vats is the same. Dibromoindigo goes on to cotton more quickly than either of the difluoroindigos. Both difluoroindigos are markedly faster to light than the dibromo derivative.

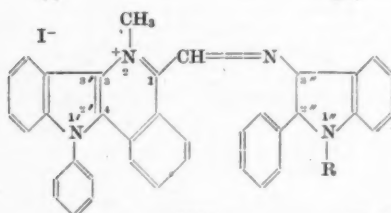
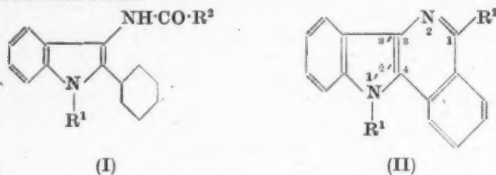
C. O. C.

1:2-Disubstituted 3-Aminoindoles. II—Preparation of Indolo(3':2'-3:4')isoquinolines and of a New Type of Cyanine Dye derived therefrom.

Huang-Hsinmin and F. G. Mann. *J.C.S.*, 2911-2914 (Nov. 1949).

The cyclisation of 3-acetamido-2-phenylindole and of 3-acetamido-2-phenyl-1-alkyl(or aryl)indoles (I) to the

1-substituted and 1:1'-disubstituted indolo(3':2'-3:4)isoquinolines (II) respectively is described. The methiodide of 1'-phenyl-1-methylindolo(3':2'-3:4)isoquinoline readily condenses with 1:2-disubstituted 3-nitrosoindoles, with *p*-nitrosodimethylaniline, and with *p*-dimethylaminobenzaldehyde to give cyanine dyes (e.g. III) of a new type. Most cyanine dyes possess two heterocyclic nitrogen atoms, each of which can accept the positive charge of the cation, and hence the dye exists as a resonance hybrid of two canonical forms; whereas the new dyes contain three heterocyclic nitrogen atoms, and thus exist in three canonical forms.



(III)

H. H. H.

Synthesis of Cyanine Dyes by the Condensation of *p*-Diethylaminobenzaldehyde with Appropriate Heterocyclic Compounds—III. M. Q. Doja and J. C. Banerjee. *J. Indian Chem. Soc.*, **26**, 374-378 (Aug. 1949).

In order to study the effect of substitution in the thiazole and benzothiazole nuclei of *p*-diethylaminostyrylthiazole and *p*-diethylaminostyrylbenzothiazole methiodides on photographic sensitisation, four new dyes have been prepared by condensing the methiodides of 2:5-dimethylthiazole and related compounds with *p*-diethylaminobenzaldehyde. The optical, chemical, dyeing, and photographic properties of these dyes are described.

A. S. F.

Colour of Pigment Mixtures. D. R. Duncan. *J. Oil & Col. Chem. Assoc.*, **32**, 296-321 (July 1949).

The laws governing the colours of pigment mixtures are explained, and their application to practical problems such as matching is discussed with reference to (a) the relative scattering and absorption coefficients of pigments and their use in predicting the colours of mixtures, (b) the range of colours obtainable from a given set of pigments, and (c) the approximate proportions of different pigments required to produce a given colour.

A. S. F.

PATENTS

2-Naphthyl-J-acid and its Urethanes—Colour Formers. V. Tulagin, W. A. Schmidt, and General Aniline & Film Corpn. *U.S.P.* 2,480,815.

2-Naphthyl-J-acid and its urethanes are stable compounds readily obtained chemically pure. Used as intermediates for dyes they produce brilliant hues showing a marked bathochromic shift in the transmission spectra compared with analogous dyes derived from phenyl-J-acid and its intermediates, and also better fastness to light and affinity for organic fibres.

C. O. C.

Dicyandiamide-Aniline Condensates—Dye Intermediates and Agents for Preventing Gas Fading of Dyed Cellulose Acetate. D. W. Jayne, Jr., H. M. Day, and American Cyanamid Co. *U.S.P.* 2,482,710.

Equimolecular proportions of an aromatic amine and dicyandiamide are heated together in presence of a little of a suitable catalyst, the excess aniline is removed, and the product is washed, dried, and pulverised.

C. O. C.

5-Acyloxypyrazole Colour Formers. Du Pont.

B.P. 632,835.

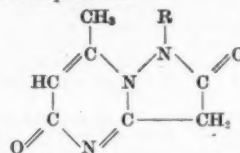
5-Acyloxypyrazole colour formers, being more stable than the corresponding 5-pyrazolones from which they are

derived, are superior to the latter in colour photography as they have less tendency for self-condensation and are more resistant to oxidation. Furthermore their use renders it possible to prepare complex colour formers containing groups which would be impractical with the 5-pyrazolones, e.g. aldehyde groups.

C. O. C.

1-Substituted 2:5-Diketo-7-methylpyrimido-pyrazoles. A. Bavley and General Aniline & Film Corpn. *U.S.P.* 2,481,466.

The reaction products of diketene with a pyrazolone having a primary NH_2 group in position 3 and a substituent on the N in position 1—



give blue images on colour-forming development despite the fact that they contain a reactive methylene group and no phenolic hydroxy group.

C. O. C.

Azo Pigments and Lakes. R. C. Loocke and Du Pont. *U.S.P.* 2,478,767.

A modification of the usual method of making azo pigments, which lends itself to the design of continuous manufacturing processes, consists in bringing together at controlled rates an acid solution of a diazotisable amine and an alkaline solution of a coupling component containing a nitrite, maintaining the reaction mass at pH 6.0-7.2 and 20-100°C. Thus a solution containing 1 mol. of 4-nitro-2-aminotoluene and 2 mol. of HCl at 38°C. and an aqueous solution containing 1 mol. of the anilide of 2:3-hydroxynaphthoic acid, 1 mol. of caustic soda, 1 mol. of sodium nitrite, and a little Turkey red oil at 65°C. are poured into a third vessel at a regular rate over 30 min., a pH of 6.0-7.2 being maintained throughout.

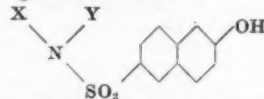
U.S.P. 2,478,768.

Azo pigment lakes are made by combining diazotisation, coupling, and laking in one operation. An acid solution containing the laking agent is mixed with an alkaline solution containing a nitrite, the diazotisable amine and the coupling component being in either of the solutions. The pH is maintained at 6.0-7.2 throughout the mixing, and temperatures of 24-100°C. are employed. Thus a solution containing HCl (2.37 mol.) and BaCl_2 (1.02 mol.) is added to a well stirred solution containing *p*-toluidine-sulphonic acid (1.0 mol.), β -naphthol (1.05 mol.), and sodium hydroxide (3.18 mol.), both solutions being at 24°C., and the temperature is raised finally to 50°C. to complete the reaction. The method lends itself to the design of continuous manufacturing processes.

E. S.

Monoazo Chrome Dyes. Ciba Ltd. *B.P.* 632,095.

Monoazo chrome dyes are made by coupling diazotised 4-chloro-2-aminophenol-6-sulphonic acid with a coupling component of general formula—



(X = aryl, substituted aryl, aralkyl, alkyl, or cycloalkyl; Y = H or alkyl). Thus by using 2-naphthol-6-sulphonamide as the coupling component, the product dyes wool brown from an acid bath, becoming blue-violet on afterchroming.

E. S.

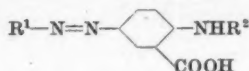
Green or Black Monoazo Chrome Dye. H. W. Grimmel, L. Richter, and General Aniline & Film Corpn. *B.P.* 632,092.

The monoazo dye 4-nitro-2-aminophenol-6-sulphonic acid-1-phthaloylamino-7-naphthol dyes wool black, converted to olive-black by afterchroming, but applied by the metachrome process it yields bright green.

E. S.

Soluble Monoazo Dyes for Cellulose Acetate, etc. from N-Substituted Anthranilic Acids. H. W. Grimmel, C. G. Vogt, and General Aniline & Film Corpn. *B.P.* 632,096.

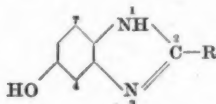
Yellow to brown soluble dyes for acetate rayon, nylon, etc. have the general formula—



(R¹ = the residue of a diazo compound free from solubilising groups; R² = alkyl or cycloalkyl free from solubilising groups). Thus by coupling diazotised 2-chloro-4-nitro-aniline with *N*-cyclohexylantranilic acid, a yellowish-brown dye is produced. E. S.

Metallised Benziminazole Monoazo Dyes. C. E. Lewis and American Cyanamid Co. U.S.P. 2,479,944.

Dyes which may be metallised in substance are made by coupling a diazo compound containing a lake-forming group *ortho* to the diazo group with a 5-hydroxybenziminazole—



(R = H, mononuclear aryl, or lower alkyl). Thus the monoazo compound 1-aryl-6-nitro-2-naphthol-4-sulphonic acid → 2-methyl-5-hydroxybenziminazole is converted into its chromium complex, to yield a grey wool dye. E. S.

Metallisable Substantive Disazo Dyes. Ciba Ltd.

B.P. 631,262.

Tetrazotised 3:3'-dihydroxy-4:4'-diaminodiphenyl is coupled with naphthols free from sulphonyl groups, but which may contain e.g. -O-C₂H₄OH, NH₂, and especially -SO₂-NH₂ groups, in presence of > 25% of pyridine on the weight of the coupling mixture, to yield metallisable substantive dyes. Purer dyes and higher yields are obtained as compared with the processes described in B.P. 609,300 (J.S.D.C., 65, 132 (March 1949)), which employ little or no pyridine. Thus the tetrazo compound from 3:3'-dihydroxy-4:4'-diaminodiphenyl is separated and added to 2-naphthol-6-sulphonamide in pyridine. Coupling is effected by slowly adding aqueous 25% caustic potash and stirring at 20°C. The mixture is diluted with water, and the pyridine distilled off under reduced pressure. The disazo compound produced dyes cellulose blue-violet by the single-bath or two-bath aftercoupling process. E. S.

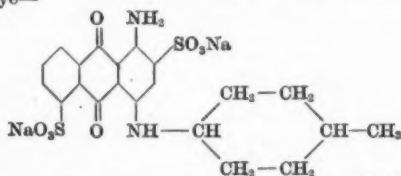
Blue Disazo Pigment. S. A. Scully and Interchemical Corp'n. U.S.P. 2,477,661.

The blue disazo pigment dianisidine-2 mol. of the 2:5-dimethoxyaniline of 2:3-dihydroxynaphthoic acid and its coppered derivative have reflectance characteristics similar to those of chlorophyll in the infra-red as well as in the visible part of the spectrum, and hence find application in camouflage paints. E. S.

Anthraquinonoid Acid Dyes. Sandoz Ltd.

B.P. 630,525.

A 1-amino-4-bromo(or chloro)anthraquinone-2-sulphonic acid carrying another sulphonyl group in the 5- or 8-position is condensed with a hydroaromatic amine, e.g. cyclohexylamine, to give blue dyes, faster to light and wet treatments than the dyes described in B.P. 276,408 (cf. J.S.D.C., 54, 28 (1928)). Alternatively the corresponding 2-bromo (or 2-chloro) derivative may be used, and the condensation product subsequently treated with aqueous alkali-metal sulphite to replace the 2-halogen by a sulphonyl group. Thus, sodium 1-amino-4-chloroanthraquinone-2:5-disulphonate is heated for 16 hr. at 60-70°C. with aqueous 4-methylcyclohexylamine, in presence of NaOH and copper powder in an atmosphere of nitrogen, to give the dye—

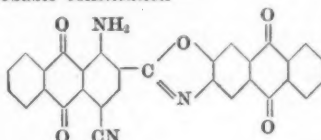


R. K. F.

Anthraquinone Derivatives—Vat Dyes. Ciba Ltd.

B.P. 630,375.

Red to brown vat dyes are made by heating a 4-cyanoanthraquinone-2-carboxylic acid (or a functional derivative), containing in the 1-position NH₂, or a substituent capable of being replaced by NH₂, e.g. NO₂, or Hal, with an amine having at least one replaceable H attached to the N, particularly an amine capable of being vatted. Thus 1-amino-4-cyanoanthraquinone-2-carboxyl chloride is heated at 150°C. with 2-amino-3-bromoanthraquinone in nitrobenzene containing cuprous chloride. After 2 hr., during which time air is passed through, fused potassium acetate and copper oxide are added, and heating at 185-195°C. is continued for a further 6 hr. The resulting dye of probable constitution—



is filtered off after cooling.

R. K. F.

Sulphuric Esters of Leuco Vat Dyes and Anthraquinonoid Intermediates. D. A. W. Fairweather and I.C.I. Ltd. B.P. 630,459.

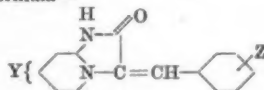
The leuco derivative, or metal salt thereof, of a vat dye or anthraquinonoid intermediate is treated with an alkali-metal, ammonium, quaternary ammonium, or ternary sulphonium chlorosulphonate in an inert anhydrous medium to give the corresponding leuco sulphuric ester. The chlorosulphonate may be formed *in situ*. Thus, methyl chlorosulphonate is stirred with dimethyl sulphate and triethylamine in acetone to form the quaternary salt. Dimethoxydibenzanthrone and iron powder are then introduced, stirring at 5-10°C. is continued for 1 hr., aqueous sodium carbonate added, and the leuco sulphuric ester isolated by salting. R. K. F.

Asymmetric Photosensitising Dyes containing a 1:3:4-Triazaindolizine (1:3:7-Triazapyrrocoline) Nucleus. General Aniline & Film Corp'n. and N. Heimbach. B.P. 632,808.

The bases of 1:3:4-triazaindolizine give rise to several different series of cyanine dyes, all of which are excellent photosensitisers. C. O. C.

Cyanine Dyes. G. Schwarz and Gevaert Co. of America Inc. U.S.P. 2,481,953.

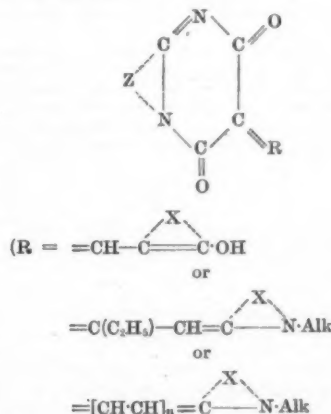
Dyes of formula—



(Y = an anion; Z = OH, O-Alk, or N-Alk₂) are useful in photographic filter or antihalation layers. C. O. C.

merocyanine Dyes. A. W. Anish and General Aniline & Film Corp'n. U.S.P. 2,482,532.

Dyes of formula—



X and Z = atoms necessary to complete a nitrogenous heterocyclic nucleus; $n = 0, 1, \text{ or } 2$ have strong photosensitising properties. C. O. C.

N-Aryloxy-, Arylthio-, and Arylseleno-alkyl-cyanine Dyes—Photographic Sensitisers. A. W. Anish and General Aniline & Film Corp'n.

U.S.P. 2,481,464.

meso-Alkoxy-substituted Symmetrical or Asymmetrical Carbocyanine Dyes—Photographic Sensitisers. Gevaert Photo-Producten N.V.

B.P. 632,638.

Cyanine and Styryl Dyes containing Two Mono- or Polymethin Chains—Photographic Sensitisers. Gevaert Photo-Producten N.V.

B.P. 632,641.

Polymethin Dyes for Antihalation and Filter Layers in Colour Photography. Gevaert Photo-Producten N.V. and L. A. van der Auwera.

B.P. 632,640.

Chlorophyll Composition for Green Camouflage Paint. K. V. Thimann and U.S. Secretary of the Navy.

U.S.P. 2,481,366.

Chlorophyll vegetable matter is ground in water, and the chlorophyll with its associated protein precipitated by adding zinc, magnesium, copper, or manganese sulphate. The precipitate is then stabilised towards light by adding sugar and magnesium sulphate, and finally partly dried. The product is used as a green pigment in camouflage paints. C. O. C.

Preheating Charge Stock for Carbon Black Furnaces to obtain Increased Yields. Phillips Petroleum Co.

B.P. 631,736.

Aluminium Phosphate Phosphor activated by Tervalent Cerium. British Thomson-Houston Co. Ltd.

B.P. 631,415.

Cadmium Red. L. F. Nerlinger and Du Pont.

U.S.P. 2,479,636.

Precipitated cadmium selenocyanide $\text{Cd}(\text{SeCN})_2$ is mixed with precipitated cadmium sulphide and then calcined. A high-quality product of optimum particle size is obtained without much loss of selenium. C. O. C.

Cadmium Sulphoselenide Pigments. F. Kertesz and Du Pont.

U.S.P. 2,482,006.

Adding 1-4% by weight of barium carbonate or peroxide before calcining the mix of cadmium sulphide, cadmium oxide, and selenium imparts uniform consistency and colour to the resulting pigment, which has also a low bulk density and is free from grit. C. O. C.

Titanium Pigments. National Titanium Pigments Ltd., J. T. Richmond, G. G. Durrant, and R. J. Wigginton.

B.P. 632,191.

The manufacture of precipitated titanium dioxide which after calcination gives a rutile pigment of tinting strength 20-60% higher than that of the usual anatase pigments is described. C. O. C.

Titanium Oxide Pigment. C. M. Olson and Du Pont.

U.S.P. 2,479,637.

An improved process for obtaining Titanium White of uniform particle size, by hydrolysis of titanium sulphate solution and calcination of the precipitate, is described. C. O. C.

Water-dispersible Titanium White. W. R. Whately and American Cyanamid Co.

U.S.P. 2,480,092.

Water-dispersible titanium dioxide of extremely fine texture is obtained by flocculating water-dispersed hydroclassified micropulverised calcined titanium dioxide by adding not > 2%, on the weight of the calcined oxide, of a hydrolysable titanium salt. C. O. C.

Fluorescent Zinc Oxide. L. J. Reimert, E. A. Fatzinger, and New Jersey Zinc Co.

U.S.P. 2,481,344.

Calcination in an inert atmosphere of zinc oxide mixed with a little lithium sulphate and a little magnesium oxide yields a brilliantly fluorescent pigment of excellent chemical and photochemical stability. C. O. C.

Finishing of Pigments. W. H. Hoback, W. J. Cauwenberg, W. R. Whately, and American Cyanamid Co.

U.S.P. 2,479,836.

Pigment particles are treated with a little of a liquid organic coating material to improve their texture, hiding power, colour, tinting strength, and gloss. C. O. C.

V—PAINTS; ENAMELS; INKS

PATENTS

Coatings on Aluminium or its Alloys. American Chemical Paint Co. and F. P. Spruance, Jr.

B.P. 632,102.

Greenish-grey to pale green coatings are obtained by treatment with an acid aqueous solution containing phosphate, fluoride, and dichromate ions, the total acidity not exceeding that of 3N. acid. They give excellent protection against atmospheric corrosion.

B.P. 632,090.

Coatings of nearly identical properties are obtained if the phosphate ions are replaced by arsenate.

B.P. 632,097.

Addition of arsenate ions to the solution improves the adhesion of the resulting coating. C. O. C.

Coatings on Iron, Zinc, and Alloys of Either. American Chemical Paint Co., F. P. Spruance, Jr., and J. H. Thirsk.

B.P. 632,099.

Firmly adherent coatings are obtained by using the solutions described in B.P. 632,102 (above). C. O. C.

Coatings on Copper, Brass, Magnesium, Iron, and Ferrous or Zinc Alloys. American Paint Co., F. P. Spruance, Jr., and J. H. Thirsk.

B.P. 632,100.

The process of B.P. 632,090 (above) is used. C. O. C.

Steam-setting Printing Ink. A. F. Schmutzler and D. F. Othmer.

U.S.P. 2,482,879.

Printing inks, which set to hard water-resistant films on treatment of the print with steam or water but which have high press-stability to moisture, contain soyabean protein not < 95% pure, ethylene or diethylene glycol, a peptising agent (preferably guanidine carbonate), a pigment, and, if desired, a resin. C. O. C.

Highly Flexible Wrinkle Coatings. New Wrinkle Inc. (III, p. 240.)

VI—FIBRES; YARNS; FABRICS

Recent Research on Cotton Properties at the Southern Regional Research Laboratories. K. Ward. *Amer. Dyestuff Rep.*, 38, P 122-P 126 (7th Feb. 1949).

This is a brief and general account of the cotton properties studied directly and includes tensile and elastic qualities, resistance to creasing, weathering, mildew, chemical attack and fire, absorbency and repellency of water, thermal and dyeing effects. H. K.

Viscosimetric Measurement of the Degree of Polymerisation of Cellulose. H. A. Wannow. *Textil-Praxis*, 4, 457-459 (Sept. 1949).

The method whereby the degree of polymerisation of cellulose is calculated from measurements of viscosity in cuprammonium solution is outlined. Similar calculations have now been made from measurements of the viscosity of regenerated cellulose fibres in sodium hydroxide solution. F. A.

Retting of Flax. F. Tobler. *Textil-Rund.*, 4, 186-190 (May 1949).

In a review of the present state of our knowledge of the retting process the differences in speed of retting not only between different types of flax but in one and the same stalk are emphasised. These differences can be readily detected by a shaking test. The circumstances under which a double retting is better than a single retting are discussed. F. A.

Action of Heat on Wool. H. Zahn. *Textil-Praxis*, 5, 7-10 (Jan. 1950).

The literature dealing with the action of heat on wool is reviewed. Wool is much more resistant to heat when dry than when moisture is present in the fibre. Predried wool can be heated to 140-150°C. for a short time without degradation taking place provided no products are present which attack wool protein. B. K.

Moisture Regain of Silk. E. A. Hutton and J. Gartside. *J. Textile Inst.*, 40, T161-T169 (March 1949).

The desorption and adsorption isotherms have been determined for Chinese, Japanese, and Italian silks both in the gum and after degumming. Vacuum and desiccator methods have been used, and the results show that degummed silks have a lower regain than the corresponding

silks in the gum at all humidities and also show less hysteresis between adsorption and desorption isotherms. The different silks adsorb water to approximately the same extent after degumming, but in the gum the Italian silk has a greater adsorption than the other types. H. K.

Supercontraction—Influence of Phenols on Wool, Horsehair, Silk, and Polyamide Fibres. E. Elöd and H. Zahn. *Melliand Textilber.*, 30, 17-23 (Jan. 1949).

According to Speakman the supercontraction of animal fibres results from rupture of cystine cross-linkages. The present authors, however, consider that supercontraction may be induced by rupture of hydrogen bonds only. The influence of phenols on silk and polyamide fibre, which do not contain cystine, was investigated. Supercontraction was observed, its magnitude being approx. constant over the pH range 1.5-9.5 and decreasing with either a more acid or a more alkaline reaction. Below pH 5 the behaviour of horsehair in phenol solution was similar to that of silk or polyamide fibre, but above pH 5 the supercontraction showed a much greater increase. It was found by measurements of cystine content that the increase in the supercontraction of keratin fibres at pH > 5 was due to simultaneous rupture of hydrogen bonds and disulphide links. The supercontraction below pH 5 was, however, due to rupture of hydrogen bonds only. F. A.

Recrystallisation of Relaxed Animal Fibres. L. Jagger and J. B. Speakman. *Nature*, 164, 190 (30th July 1949).

Restrengthening of human hair weakened by stretching and holding at 30% extension is slow in water at ordinary temperatures, but is accelerated by raising the temperature. Maximum recovery is obtained at 70°C. The results indicate that the beneficial effects of ageing processes in the manufacture of wool textile materials may be obtained in much shorter times. G. L.

Fibrous Proteins—Classification of Fibrous Proteins. I—K. H. Meyer; II—W. T. Astbury. *Nature*, 164, 34-36 (2nd July 1949).

I—Astbury's classification of fibrous proteins into two groups, the collagen group and the keratin-myosin-fibrinogen group, the latter exhibiting "long-range" elasticity attributable to attractive forces tending to fold the protein chains in the contracted state, is contested by Meyer, who maintains that "long-range" elasticity is not limited to the keratin-myosin-fibrinogen group, since all fibrous proteins may be brought into the rubberlike state by heat treatment or plasticisation.

II—Astbury maintains that the free energy is divided into two portions, part being allotted to the restoring force and internal energy effects and the remainder to entropy effects. Woods and Bull have estimated the two parts separately. With rubber the entropy factor is large compared with the internal energy factor, whilst the converse is true for normal animal hairs, indicating that normal keratin does not exhibit the elastic properties of a true elastomer. A reply is also given to Meyer's criticism of Astbury's X-ray data. H. K.

Fibrous Proteins—Rubberlike Properties of Hair Keratin. I—K. H. Meyer and C. Haselbach; II—H. J. Woods. *Nature*, 164, 33-34 (2nd July 1949).

I—From a study of the thermoelastic properties under small torsions of hair swollen in 70% thioglycolic acid, vulcanised rubber, and plasticised polyvinyl chloride, it is concluded that plasticised hair behaves in exactly the same way with respect to entropy and free energy changes at small torsions as do true elastomers or plasticised potential elastomers, and that the elastic restoring force of swollen hair keratin is of the same nature as that of true elastomers; i.e. the restoring force is due to thermal movements of the chain segments, as is shown by the increase in entropy on restoration of the original state. This is in contrast with the theory of Astbury and Woods that the restoring force of stretched keratin can be attributed to attractive forces, which favour a folded configuration of the keratin protein chains.

II—Woods maintains that the theory of Meyer and Haselbach is unreliable, since keratin is a cross-linked, oriented structure, and that thioglycolic acid treatment causes disulphide linkage breakdown and swelling, of such an order that there is considerable loss of birefringence and

crystallinity. The polypeptide chains are thus so far isolated that the appearance of rubberlike properties is not surprising. The fibre is so far modified, however, as to make the results of little value in the study of the elastic mechanism in normal fibres. H. K.

Structure of Natural and Synthetic Fibres. H. Staudinger. *Textil-Rund.*, 4, 3-17 (Jan. 1949).

A general review of fibre structure is given with special reference to the determination of molecular weight and the relation between degree of polymerisation and fibre properties. The chemical changes resulting from oxidation of cellulose fibres are outlined. The phenomenon of the inclusion of organic substances such as benzene and carbon tetrachloride in fibres is described. F. A.

Cuprammonium Solutions of Cellulose. II—Effect of Ammonia on the Absorption of Copper and on the Solubility of Cellulose in Cuprammonium Solution. M. I. Arkhipov and V. P. Kharitonova. *J. Appl. Chem. (U.S.S.R.)*, 22, 1030-1036 (Sept. 1949).

Determinations were made of the absorption of copper by cellulose from cuprammonium hydroxide preparations in which the concn. of ammonia was varied within wide limits. The copper content was also varied, but the composition was always such that no appreciable dissolution of cellulose occurred. As the concn. of ammonia increased from a low value at constant copper content, copper absorption fell, passed through a minimum, and then rose. The higher the copper content, the greater was the concn. of ammonia at the point of minimum absorption. The results are analogous to those given for optical rotatory power in the first paper of the series (cf. *J.S.D.C.*, 66, 79 (Jan. 1950)) and are explained on the same lines.

Determinations were also made of the solubility of cellulose in a range of cuprammonium hydroxide preparations. The copper concn. range was higher than in the absorption experiments. Other factors being constant, the higher the ammonia content, the higher the solubility. It is considered that the main factors determining solubility are the degree of combination of cellulose with copper and the efficiency of the medium as a solvating agent. A. E. S.

X-Ray Irradiation of Cellulose Acetate. N. N. Winogradoff. *Nature*, 165, 72 (14th Jan. 1950).

Samples of undyed, uncoiled cellulose acetate yarn exposed to X-rays for periods up to 120 hr. decreased in tensile strength, measured at 65% R.H. and 20°C., by > 50%. Similar results were obtained with a yarn containing 3% titanium dioxide delustrant. All breakages occurred within the limits of the irradiated patch, and under a microscope showed a very brittle fracture; the strengths remained constant between 120 and 220 hr. exposure. X-Ray irradiation is found also to decrease the crystallinity of the sample. When a strip of clear unplasticised cellulose acetate film was covered by a layer of wax and then irradiated for 24 hr., bubbles of gas occurred in the wax which forced it away from the film. These chemical and structural changes may influence the interpretation of X-ray diagrams. H. H. H.

Modes of Swelling and Solution of Fibrous Nitrocellulose of High Nitrogen Content. W. R. Moore. *J. Textile Inst.*, 40, T731-T733 (Oct. 1949).

Several liquids and binary liquid mixtures have been classified as solvents and non-solvents for nitrocelluloses of high N content. Solvents may act either by a process termed "immediate gelation", in which the fibres immediately become structureless masses, or by a slower process involving breaking of fibres into elongated fragments and eventual dispersion. In the case of solvent-non-solvent mixtures, three factors influence the degree and mode of swelling—(a) the solvent used with a given non-solvent, (b) the fibre, and (c) the non-solvent used with a given solvent. Both types of solution process are interpreted in terms of fibre structure and molecular size of the solvent. A. S. F.

Denaturation in Regenerated Protein Fibres. F. Happey. *Nature*, 164, 184-185 (30th July 1949).

The X-ray photograph of highly oriented casein fibres consists of the normal β -protein type of diagram and a polar arc characteristic of the cross β -pattern, showing the presence of a two-phase system thought to arise from partial denaturation during solution. It is suggested that

a similar system could explain Mercer's finding that the α -form of the wool molecule is retained in regenerated keratin fibre. The α -structure could not be identified in more recent work on regeneration of wool. G. L.

Adsorption and Desorption of Water by Nylon at 25°C. E. A. Hutton and J. Gartside. *J. Textile Inst.*, 40, T170-T174 (March 1949).

The adsorption and desorption isotherms for nylon 66 show a small but definite hysteresis effect. Adsorption decreases with increasing temperature of drying, but the normal affinity for water is largely restored after saturation with water vapour. The regain at saturation is 8.7%.

Moisture Absorption, Density, and Swelling Properties of Nylon Filaments. N. J. Abbott and A. C. Goodings. *J. Textile Inst.*, 40, T232-T246 (April 1949).

Moisture absorption of nylon may be either increased or decreased by treatment with certain reagents. Moisture absorption isotherms for drawn and undrawn nylon filaments have been determined; effect of pretreatment with alcohol has been studied up to 90% R.H. Appreciable differences in regain of these samples occurs only in the upper half of the R.H. range. Densities of drawn and undrawn nylon filaments in the R.H. range 0-98% have been measured by a flotation technique. Apparent densities in water have also been determined. A length contraction occurs on first wetting; moisture subsequently produces longitudinal swelling, the magnitude of which has been measured between 0 and 100% R.H. Corresponding volume changes and lateral swelling have been computed. In drawn nylon longitudinal and lateral % swellings have been found to be approx. equal, but in undrawn nylon longitudinal swelling is three times the lateral swelling. A major portion of the empty space in nylon filament is not penetrated by water. G. L.

Low Temperature Properties of Textile Materials. E. R. Kaswell. *Amer. Dyestuff Rep.*, 38, P 127-P 134 (7th Feb. 1949).

A study has been made of the performance of yarns likely to be used in parachute suspension lines at -70°F . The load-extension and energy absorption characteristics of silk, Fibre A, Vinyon E, and various types of nylon have been compared at 70°F . and -70°F ., along with the difference in effect produced by "shock" and "slow" loading. H. K.

PATENTS

Recovering Fur, Hair, or Wool from Hide Scraps and Skins. M. Mulqueen and Hatters Fur Exchange Inc. U.S.P. 2,480,761.

The materials are treated with a 0.1-1% solution of pepsin in 0.5-3.0 N-HCl until the hide dissolves, after which the fibres are separated from the solution. C. O. C.

Alginate Threads resistant to Alkali. Alginate Industries Ltd., J. B. Speakman, N. H. Chamberlain, and C. M. C. Dorkin. B.P. 631,002.

The alkali resistance of alginic acid derivatives is improved by swelling in water, displacing the water with methanol, and then cross-linking by treatment with an organic diisocyanate for 24 hr. W. G. C.

Protein Rayon. D. Traill, E. L. Thomas, and I.C.I. Ltd. B.P. 632,240.

Viscous alkaline peanut globulin solutions are extruded into an acid saline solution and the coagulated filaments passed, before insolubilising, into one or more non-swelling saline solutions at not $> 40^{\circ}\text{C}$., in which the filaments are led on to and pressed against one or more supporting surfaces. At least that saline solution preceding the first supporting surface includes 0.1-2% by weight of a cationic surface-active agent, e.g. cetyltrimethylammonium bromide. J. W. B.

Polyamides containing a Sulphone Grouping. British Celanese Ltd. B.P. 631,020.

Fibre-forming polyamides are obtained by treating a dicarboxylic acid containing a sulphonyl group, e.g. sulphonyl- $\delta\delta'$ -divaleric acid, with equimol. quantities of a polyamine having two of its amino groups primary and the remainder secondary, e.g. 1:9-diamino-5-azanonane. W. G. C.

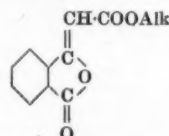
Linear Polyesters. Wingfoot Corp. B.P. 630,992.

Fibre-forming polyesters are obtained by treating glycols containing $> 4\text{C}$ with equimol. quantities of terephthaloyl and isophthaloyl chlorides until the mol. wt. is $> 10,000$. W. G. C.

Solutions of Vinylidene Copolymers. Société Rhodiacéta. B.P. 631,055.

Copolymers of vinylidene chloride with unsaturated esters and with vinyl halides dissolve in organic liquids containing 9-70% carbon disulphide to give solutions which are useful for fibres, films, coatings, varnishes, etc. W. G. C.

Copolymers of Esters of Phthalideneacetic Acid with Styrene—Fibre-forming Materials. D. T. Mowry and Monsanto Chemical Co. U.S.P. 2,483,371. Compounds of formula—



can be polymerised, especially in presence of more active monomers. Their copolymers with styrene are valuable fibre-forming materials, as they are subject to less heat distortion than polystyrene. C. O. C.

Fibres from Rubber-like Materials. N. V. De Bataafsche Petroleum Maatschappij. B.P. 608,078.

This specification has been amended, but not so as to affect the abstract previously published (J.S.D.C., 65, 87 (Feb. 1949)). C. O. C.

Relation of Physical Characteristics and Chemical Structure of Cellulose Derivatives. E. Ott. (XIII, p. 252.)

Stabilising Polyethylene. Du Pont. (XIII, p. 252.)

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Practical Implications of the Properties of Enzyme Desizing Agents. E. Schubert. *Textil-Rund.*, 5, 1-14 (Jan. 1950).

The various types of amylases which act as desizing agents are described. Graphs are given to show the effect of temp., liquor ratio, stabilisers, and pH on the activity and stability of desizing agents. Desizing tests are discussed, and the factors influencing bulk desizing are given as the type and % of amylase, composition of desizing bath (stabiliser, etc.), liquor ratio, temp., pH, time of treatment, type of material to be desized, wetting properties of liquor, and method of desizing. B. K.

Internal Wetting of Fibres as a Factor in Detergency. J. Powney. *J. Textile Inst.*, 40, T 519-T 523 (Aug. 1949).

"Complete wetting out" is generally accepted as meaning the penetration of the solution into the spaces between the yarns of the fabric and also into the smaller interstices between the individual fibres of each yarn. Experiments, originally conducted to examine the contours of oil drops when solutions of different interfacial tension were used, indicated that air bubbles were displaced from the internal regions of the fibre into the oil drops. It is thought that penetration takes place on either side of the oil drop, which cannot penetrate the fibre. Air locks are thus set up which release their pressure only through the oil-covered capillary orifices of the fibre. The effect is interesting in that it is a well known fact that certain types of resistant oil are not removed by normal washing treatments but can be removed by a prolonged soaking process. H. K.

Low Temperature Wool Piece Goods Scouring with a Synthetic Organic Detergent—Laboratory Evaluation. O. M. Morgan and J. E. Walter. *Amer. Dyestuff Rep.*, 38, P 374-P 377 (2nd May 1949).

The scouring of woollen piece-goods containing 5-7% emulsified mineral oil by Naeconol NR [alkarylsulphonate] in the presence of builders has been compared at 80°F . and at 120°F . under laboratory conditions. Exhaustion of the scouring bath was studied by treating six consecutive

patterns in the same solution. At 80°F. satisfactory scouring (< 1% residual oil) was obtained by the use of 0.3–0.4% solution of detergent along with 1% solution of sodium chloride or 0.4% detergent and 1% sodium bicarbonate, whereas at 120°F. 0.2% detergent with the same quantities of builders was sufficient. Sodium carbonate was generally found to be an inefficient builder at both temperatures. It is considered that in spite of increased chemical costs the savings in steam, time, etc. obtained by scouring at low temperatures will result in overall economy.

W. K. R.

Desoiling Properties of Carboxymethyl Cellulose. A. J. Feuill. *J. Textile Inst.*, 40, T 523–T 525 (Aug. 1949).

Sodium carboxymethyl cellulose has been found to enhance slightly the desoiling power of Lissapol N, a non-ionic type of detergent, although it does not itself possess soil removal properties and does not appear to function as a simple "builder".

H. K.

Comparative Efficiencies of Wetting Agents on Woollen Cloth. K. S. La Fleur. *Amer. Dyestuff Rep.*, 38, 367–368 (2nd May 1949).

The comparative efficiencies in wetting out woollen cloth of 21 commercial wetting agents of different chemical types, comprising anionic, cationic, and non-ionic products, have been determined. Results are expressed in terms of both concentration (of active ingredient) and cost of product required to produce a sinking time of 10 sec. at room temp. On a concentration basis, sodium dioctyl sulphosuccinate was easily the most effective, followed by (in order of decreasing efficiency) non-ionic condensates of the polyethylene ether type, sodium salts of secondary alkyl sulphates, alkyl- and alkaryl-sulphonates, primary alkyl sulphates, fatty acid amide and ester sulphonates. On a cost basis, however, the polyethylene condensates were somewhat superior to sodium dioctyl sulphosuccinate.

W. K. R.

Continuous Bleaching of Cotton. A. Klein. *Textex*, 14, 5–21 (Jan.), 75–83 (Feb.), 95–103 (March), 143–149 (April), 193–205 (May), 237–249 (June), 289–299 (July), 325–331 (Aug.), and 369–375 (Sept. 1949).

Arguments are presented in favour of bleaching cotton goods by a continuous method. This type of process is then discussed in a practical manner with reference to the types of machines to be employed. The bibliography contains 131 items.

F. A.

Methods and Reagents used in getting the Colour Off. A. G. Tyler. *Dyer*, 103, 23–25, 27 (13th Jan.) and 154 (10th Feb. 1950).

Recipes for stripping the various classes of dye from textiles are given, the methods and degree of stripping required depending upon the subsequent treatment which the materials are to undergo.

H. K.

PATENTS

Simultaneously Scouring and Bleaching Wool. A. L. Dubeau and Mathieson Chemical Corpn.

U.S.P. 2,481,205.

An aqueous liquor containing sodium carbonate and bicarbonate, soap, and sodium chlorite is used, the weight ratio of apparent carbonate to bicarbonate being kept at 1:0.5–2.0.

C. O. C.

Bleaching of Paper Pulp. R. M. Levy, J. E. Brennan, and Ecusta Paper Corpn.

U.S.P. 2,480,148.

The viscosity and the copper number are both kept at a high value if bleaching is done with an alkaline bleach solution, e.g. sodium hypochlorite, containing a water-soluble cobalt or nickel salt as catalyst.

C. O. C.

Flax Wet Spinning Frame as a Source of Catalytic Damage in Bleaching. W. Honneyman. (XIV, p. 255.)

VIII—DYEING

Significance of rH Values in Textile Processing. A. Schaeffer. *Melliand Textilber.*, 30, 111–115 (March 1949).

The derivation, significance, and measurement of rH values are described. The rH values of sodium hydro-sulphite and Rongalite solutions at different concentrations, temperatures, pH values, and after various times of boiling are given.

F. A.

Effect of Metals on Tippy Dyeing. H. E. Millson. *Amer. Dyestuff Rep.*, 38, P 378–P 392 (2nd May 1949).

The influence of traces of other metals, particularly Fe, Cu, Al, Co, and Ni, present in the dyebath during the dyeing of wool with chrome dyes, has been studied. These metals may be derived from trace impurities in dyes and auxiliaries, from machinery, but chiefly from the water supply. They are particularly detrimental in afterchrome and chrome-mordant dyeing, because they form metal-dye complexes more rapidly and at lower temperatures than chromium. The undesired metal is not replaced by subsequent afterchroming except in the case of aluminium, where partial replacement occurs. With many dyes, the presence in the dye liquor of < 1 p.p.m. of interfering metal causes a visually perceptible change in hue, and it is calculated that in dyeing at 60:1 liquor: goods ratio with 1% of a typical dye, 1 p.p.m. iron will convert 10%, and 1 p.p.m. aluminium will convert 20%, of the dye into the respective metal complex. It is shown by microscopic examination that the dye is metallised preferentially in the wool fibre tips and other damaged parts; subsequent chroming causes little change of hue in the tips, but the body portions of the fibre acquire the correct chromed hue. Radio-autographs obtained from dyeings carried out in the presence of traces of uranium and radioactive cobalt confirm the preferential absorption of metal in the tips. In the absence of interfering metals, chromium is similarly preferentially absorbed by the fibre tips, and this unequal absorption of chromium results in tippy dyeing. Wool dyed afterchrome in the presence of 2 p.p.m. of impurity metal had generally inferior fastness to wet treatments, and in some instances to light.

W. K. R.

Wool and Fur Felt Hat Dyeing. H. J. Hall. *Dyer*, 102, 23–27 (15th July 1949).

A review of the methods for forming, hardening, and milling fur and wool felt hat cones, including dyeing with acid levelling and milling dyes. The Freeman-Taylor Crescent and the Thompson Bros. hood dyeing machines are described.

H. K.

Absorption of Acid and Substantive Dyes by Wool, Silk, Ardlid, and Nylon. E. Elöd and H. G. Fröhlich. *Melliand Textilber.*, 30, 579–585 (Dec. 1949).

It is shown that the absorption of acid dyes at pH 1.0–2.0 is based on the existence of an electrostatic linkage between the charged amino groups of the protein fibre and the dye anions, while in the isoelectric region besides sorption processes especially formation of hydrogen bridges between dye and fibre must be taken into consideration. When dyeing polyamides below pH 2, the greatly enhanced absorption of dye is brought about by linkage of the dye to the carboxamide group, which has become positively charged by taking up protons. The above processes play an important part also in the application of substantive dyes. In this case, however, dyeings carried out at ordinary temperatures have shown that, apart from chemical structure, prime importance has to be attached to histological structure of the fibre (scales, intermediate membrane layer, etc.).

B. K.

Effect of the Carboxyl Groups in Viscose Sheet on the Equilibrium Absorption of Chrysophenine G. H. A. Standing and J. O. Warwicker. *J. Textile Inst.*, 40, T 175–T 188 (March 1949).

When viscose sheet was dyed to equilibrium with Chrysophenine G in solutions containing low concentrations of sodium chloride, the equilibrium absorption was found to depend on the ratio of the mass of the viscose sheet to the volume of the dye solution. The effect was greatest when the sodium chloride concentration was low and was due to a lowering of dyebath pH caused by the presence of acid carboxyl groups in the cellulose sheet. The theory is discussed and it is suggested that the number of carboxyl ions which directly affect the equilibrium absorption may be less than the total number of carboxyl groups in the viscose sheet.

H. K.

Replica Studies of Dyed Nylon. F. A. Hamm and J. J. Comer. *Anal. Chem.*, 20, 861–870 (Sept. 1948).

"Shadowed" thin-film (100–150 Å.) sublimed silica replicates of nylon dyed with Indanthrene Golden Yellow IGK, Brilliant Pink IR, and Brown IRRD were prepared, in which the nylon was dissolved away but the dye particles were left adhering to the silica. These replicates

were prepared from dyeings before and after steaming. Light-microscope examination of the actual dyeings reveals only vaguely an increase in dye particle size, due to steaming, in all three cases, and indicates some large particles after steaming, of length $17\ \mu$. Electron-microscope examination of the replicates reveals the considerable growth in dye-crystal size on steaming, especially for the pink dye, the dimension of $17\ \mu$ for some crystals being confirmed. The mechanism suggested for this change is recrystallisation after dissolution in the nylon. The loss of tinctorial strength and increased rubbing-off tendency observed after steaming are both explicable in terms of the increase in particle size with associated migration of dye to the surface; also the observed increased stability to ultraviolet radiation after steaming is explicable on the assumption that photochemical decomposition of the dye is a surface effect, the particle surface decreasing with increase in particle size. The silica replicates were also subjected to electron-diffraction examination. The spacings of the resulting ring patterns agree well with those from pure dye specimens, suggesting a method of identifying dyes on fabrics via the silica replicate technique which would require only a few milligrams of fabric and a few micrograms of dye. The extension of this technique to other synthetic textile fibres is being studied. H. I. S.

Application of Acid Dyes to Nylon. J. P. Niederhauser. *Teintex*, 14, 407-429 (Oct. 1949).

The theory underlying the behaviour of acid dyes on nylon is discussed. For practical purposes acid dyes are divided into three groups, which are applied from strongly acid, weakly acid, and neutral baths respectively. Recommendations are made for the choice of dyes to be used in mixtures and for the production of both solid shades and reserved effects on wool-nylon mixtures. F. A.

Dyeing Behaviour of Polyamides. IV—Absorption of Acid and Direct Dyes and Strong Acids. E. Elöd and H. G. Fröhlich. *Melliand Textilber.*, 30, 103-106 (March 1949).

The absorption of HCl by nylon in the pH region 1-4 has been measured. The results agree qualitatively with those given by Carlene, Fern, and Vickerstaff (J.S.D.C., 63, 388 (Dec. 1947)) in showing a large increase in acid-binding capacity below pH 2.3, but disagree in that the acid absorption at the point of inflection was found to be 0.05 rather than 0.035 m-equiv. per gram of nylon. The difference may be due to differences in the degree of polymerisation of the nylon used. The acid absorption of 0.05, assuming saturation of primary amino groups, corresponded to a mol. wt. of 11,000-17,000 of the nylon used, and this agreed with viscosimetric measurements. The swelling of nylon in the pH region 0-7 was a minimum at pH 2-3, i.e. that at which the point of inflection in the acid-binding capacity curve occurs. A series of polyamide fibres was degraded by treatment for various times in 0.05 N-HCl at 90°C. The molecular weights of the degradation products as calculated from measurements of viscosity, dye absorption, and acid absorption were in good agreement. The swelling of the fibres increased with decrease in mol. wt. The equilibrium absorption of a number of acid and direct dyes on nylon is plotted as a function of pH. All the dyes behaved in a similar manner, showing approx. constant absorption in the pH region 2-5 and a gradually decreasing absorption at higher pH, almost no dye being absorbed at pH 9. Calculation of mol. wt. from dye absorption measurements agreed with viscosimetric determinations for a series of different polyamide fibres. F. A.

Dyeing of Perlon with Vat Dyes. J. Müller. *Melliand Textilber.*, 30, 106-110, 147-152, 199-201 (March, April, May 1949).

Perlon (polyamide) fibre can be dyed with vat dyes using the usual recipes but dyeing at 80-90°C. or, in the case of presteamed material, 60°C. Air oxidation is not sufficient; 2 g. sodium perborate or percarbonate per litre is used at 80°C. A detailed account is given of the behaviour of various classes of vat dye on Perlon, and the light, washing, and rubbing fastness properties of a large range of vat dyes on this fibre and on viscose rayon are compared. The differences between the colour of the dyes on Perlon and Vistra are described, and data given on the absorption of the dyes on these two fibres. The influence of temperature on absorption is more marked with Perlon than with

Vistra. When dyeing the two fibres in one bath it is possible to obtain solidity of shade by suitable choice of dyes and by manipulation of the temperature. F. A.

Dyeing Polyurethane Fibres. E. Köster. *Textilpraxis*, 4, 390-392 (Aug. 1949).

Polyurethane fibre (Perlon U) has less affinity for dyes than polyamide fibres (Perlons L and T) but can be dyed with Celliton and Celliton Fast dyes. The fastness properties of the more important members of this range of dyes on Perlon U are listed. Polyurethane fibre does not show any acid-binding capacity at pH $> \sim 2.3$, but acid dyes can be applied in presence of 25-50 c.c. conc. HCl or 10-20 c.c. conc. H_2SO_4 per litre. Fastness properties are given for a selection of acid dyes applied in this way. The acid concentration of the dyebath can be decreased to about one-quarter if the material is first steamed for 15-20 min. under 1-1.5 atm. excess pressure. Afterchrome dyes have been applied in a similar manner to the acid dyes. Vat dyes can be applied at 90-95°C., and basic dyes in presence of swelling agents. F. A.

Dyeing of "Orlon" Acrylic Fibre and its Use in Coloured Textiles. R. J. Thomas and P. J. Meunier. *Amer. Dyestuff Rep.*, 38, P 925-P 932 (12th Dec. 1949).

Acid, chrome, direct, sulphur, and azoic dyes have little affinity for polyacrylonitrile continuous filament yarn. The fibre can be dyed by prolonged boiling in dispersed acetate dyes, but exhaustion is poor. Heavier shades with acetate dyes have been produced by dyeing under pressure above 212°F. or by addition to the dyebath of organic assistants such as *m*-cresol, aniline, resorcinol, and anthranilic acid. The anthraquinone vat dyes have virtually no affinity for the fibre, but indigo and thioindigoid types can be dyed successfully, especially with high concentrations of potassium carbonate in the dyebath. Prints with acetate, basic, and vat dyes have good fastness to washing and light but relatively poor fastness to rubbing. It is thought that resin-bonded pigments are likely to be used extensively on Orlon. H. K.

PATENTS

Dyeing Cellulose Acetate. Textron Inc. B.P. 632,938.

Addition of inorganic acids to alcoholic dyebaths appreciably arrests the action of the latter upon the cellulose acetate and has the same effect on the action of any organic solvents present in the bath. Addition of organic acid solvents of cellulose acetate enhances the dyeing activity of alcoholic dye liquors. These effects are made use of in the application of acid dyes to cellulose acetate by using liquors containing more water than alcohol, the necessary acidity being obtained with an inorganic and/or organic acid. The concentration of the components should be such that the solution is between its threshold value (i.e. that at which it shows markedly greater wetting and penetrative capacity for cellulose acetate than the corresponding mixture of water and alcohol alone) and its critical value (i.e. that at which marked increase in absorption occurs). Deep to very deep dyeings of true tone and improved fastness are obtained without serious shrinkage or other deleterious effect on the cellulose acetate. C. O. C.

Coloration of Polyamide Fibres. A. Butterworth and P. F. Crosland. B.P. 632,083.

The fastness to light of many vat dyeings on nylon is increased by treating them with an aqueous solution which contains one or more benzene derivatives containing a hydroxy and/or carboxy group and which has no destructive swelling action on the nylon, e.g. cresol, phenol, salicylic acid, *m*- or *p*-hydroxybenzoic acid, cinnamic acid, or β -phenylpropionic or β -phenylpropionic acid. C. O. C.

Dyeing Leather, Leather Substitutes, Suèdes, and Heavy Textiles. G. R. B. Taylor. B.P. 631,928.

Dyeings lighter than the original dyeings are produced by immersing the materials in or brushing or spraying them with a composition consisting of methylated spirit, spirit dye, nitrobenzene, and printers' or spirit white. C. O. C.

Colouring Oxide-coated Aluminium or its Alloys. Aluminum Co. of America. B.P. 631,829.

By treating at 120-140°F. with an aqueous 5-10% ferric oxalate solution adjusted to pH 6.0-6.5 by addition of an alkali-metal or ammonium oxalate, a wide range of colours

from light golden yellows to dark reddish browns may be obtained. They are permanently resistant to fading on exposure to light and have excellent resistance to weathering.

C. O. C.

IX—PRINTING

Reducing Properties of Vegetable Thickening Agents.

R. Haller. *Textil-Praxis*, 4, 460-461 (Sept. 1949).

The author's method for estimating the reducing power of modified starches, by heating their solutions with caustic soda and Cibacron Yellow GN and observing the extent of colour change, has been applied to a series of thickening agents. The reaction was nil with cellulose ether, weak with farina, marked with wheat and maize starches, and strong with British Gum. When prints of Cibacron Yellow GN were made in the absence of the usual reducing agents, it was found that the amounts of dye fixed did not correspond with the reducing powers of the thickeners. More dye was fixed with cellulose ether than with British Gum.

F. A.

Problems in Ageing Textile Prints and their Control.

P. J. Choquette and O. F. Habel. *Amer. Dyestuff Rep.*, 38, P 919-P 922 (12th Dec. 1949).

Pilot plant tests have shown that relative humidity, temperature in the ager, quality and rate of flow of steam, and volume of air in the ager are important factors to be controlled in the ageing of prints.

H. K.

PATENTS

Stabilisation of Printing Pastes containing Diazonium Salts.

F. H. Adams and American Cyanamid Co. U.S.P. 2,479,890.

Thinning out of the printing paste is avoided if the diazonium salt, either in stabilised form or freshly precipitated, is associated with a water-soluble acid amide having at least one amide hydrogen replaced by an organic radical containing at least one olefinic double or acetylenic triple bond, e.g. *N*-allylacetamide.

C. O. C.

Diazotype Printing on Textiles.

General Aniline & Film Corp. and S. C. Slikin. B.P. 632,932.

The cloth is impregnated with a diazo compound which is decomposed by light to form a non-coupling compound, an organic compound which couples in alkaline medium with the unexposed diazo compound, and 4-10% ZnCl₂ on the weight of the two components exclusive of any needed to form a complex salt with the diazo compound. It is then exposed to light and treated with an alkaline developer. Thus viscose rayon, polyvinyl chloride, or cotton cloth is impregnated with a mixture of a *p*-alkyl-aminoaryldiazo compound, 2,3-dihydroxynaphthalene, and the requisite ZnCl₂. It is then exposed to light through a superimposed pattern, and finally treated with ammonia under pressure.

C. O. C.

Decorating Articles having Synthetic Resin Surfaces.

Dunlop Rubber Co. Ltd. and M. A. Harper. B.P. 631,850.

A powder containing the same synthetic resin as the surface is applied in presence of a solvent.

C. O. C.

Preparing Aluminium Foil for Bonding, Printing, and Colouring.

F. L. Eichner, and Reynolds Metals Co. U.S.P. 2,480,455.

Unevenness, sticky edges, and wrinkles are avoided if the coiled foil is heated to an annealing temp., cooled to not < 100°r., uncoiled at that temperature, and immediately bonded, printed, or coloured.

C. O. C.

Polyvinylacetal Colour Formers.

O. W. Murray and Du Pont. U.S.P. 2,481,476.

At least one stratum of a colour photography element consists of a hardened polyvinylacetal colour former capable of forming quinoneimine or azomethine dyes.

C. O. C.

Coloration of Polyamide Fibres.

A. Butterworth and P. F. Crosland. (VIII, p. 248.)

the size may be applied under laboratory conditions, and tests for determining the adhesion of the size and for measuring the stretch and recovery, firmness, and loop-forming properties of sized yarn, are described. W. K. R.

Recent Experiments in Cloth Finishing.

C. S. Whewell, A. Charlesworth, and R. L. Kitchin. *J. Textile Inst.*, 40, p 769-p 783 (Aug. 1949).

The effect of pH on the setting of wool cloths at the boil has been investigated; optimum conditions are at pH 7. Results on scouring of wool containing blends of mineral oil and polar compounds are reported. In a study of shrink-resistance quantitative data are provided on the rate and amount of chlorine absorption which occurs at various pH values. Quantitative aspects of raising involving variation of cloth structure and effect of chemical treatment are discussed.

G. L.

Treatment of Cellulosic Fibres with Hexanedio Bischloromethyl Ether.

H. Wenderoth. *Melliand Textilber.*, 30, 205-207 (May 1949).

The wet strength of cellulosic rayons is increased by treatment with bifunctional chloromethyl ethers. A simplified method for the preparation of hexamethylenebis(oxyethylpyridinium chloride) is described. This compound along with sodium acetate is padded on to the material, which is then dried and heated to 130-140°c. for a few min. By this treatment the wet strength of the material can be increased 30-40%, the swelling reduced from 90% to 45-50%, and the alkali solubility reduced from 30% to 1-5%. The influence of the temperature of treatment on these properties is given. The treated material has only a low solubility (4-8%) in cuprammonium hydroxide solution.

F. A.

Deterioration of Cotton Textiles exposed to the Weather.

H. Bogaty. *Amer. Dyestuff Rep.*, 38, 253-259 (21st March 1949).

Tensile strength and cuprammonium fluidity measurements have been made on fabrics exposed to weathering after impregnation with solutions of copper naphthenate, copper sulphate, phenyl salicylate, pyrogallol, quinol, α -naphthol, and phenyl naphthenate. The results are tabulated and show that more knowledge is required of the individual factors which comprise "weathering".

H. K.

New Method of Forming Vinyl Polymers in Wool.

M. Lipson. *Nature*, 164, 576 (1st Oct. 1949).

In the polymerisation of vinyl compounds in wool it is usual to introduce an external form of initiator such as iron (Fe⁺⁺) and hydrogen peroxide. It has been discovered that the wool can supply its own catalyst in the form of reduced cystine. (i) A conditioned sample (2.5 g.) of purified all-wool flannel was immersed in 30 ml. of a 10% solution of thiolacetic acid for 3 hr. at 30°c. (ii) A second sample was placed in 50 ml. of 2% sodium bisulphite solution, raised to the boil in 5 min., and boiled for 3 min. (iii) A third sample was similarly treated with a 2% solution of sodium hydrosulphite. After treatment, each was washed in running water, and then immersed in 50 ml. of a 4% solution of methacrylic acid for 16 hr. at 22°c. After washing for 8 hr. in running water, the samples were conditioned and weighed. Increases in weight due to polymer deposition were (i) 14.5%, (ii) 10.7%, and (iii) 3.1%. Untreated wool did not show any significant increase in weight. Pretreatments with thiolacetic acid and sodium bisulphite are each very effective in promoting polymerisation.

H. K.

Application of Synthetic Resins to Wool.

M. Lipson. *Textile J. of Australia*, 24, 566-572 (20th Aug. 1949).

Recent researches on formation of condensation and addition resins in wool fabrics are reviewed. It is reported that melamine-formaldehyde resins may not, contrary to earlier belief, penetrate the wool fibre, and that shrinkage resistance may be due to interfibre binding. Some details are given of the method of forming vinyl polymers internally, employing free-radical initiation obtained from a ferrous salt and hydrogen peroxide.

G. L.

Mothproofing.

R. Zinkernagel. *Textil-Rund.*, 4, 169-182, 212-216 (May, June 1949).

In a biological section the habits of the insects which attack wool are described. A history of the development of protective agents is followed by a review of the methods used for their evaluation.

F. A.

X—SIZING AND FINISHING

Laboratory Evaluation of Sizing Materials for Nylon Hosiery Yarns.

H. B. Goldstein. *Amer. Dyestuff Rep.*, 38, P 372-P 373, P 377 (2nd May 1949).

The sizing of nylon hosiery yarns and the requirements of a satisfactory size are discussed. A method by which

Fungicide-treated Cotton Fabric. S. S. Block. *Ind. Eng. Chem.*, 41, 1783-1789 (Aug. 1949).

Samples of washed and solvent-extracted 10-oz. cotton duck were treated with various fungicides in 1% concn. on the dry weight of fabric. Outdoor exposure tests were carried out in the shade for two years. The copper and silver compounds afforded a high degree of protection as judged by tensile strength tests. In these tests the loss in strength of the untreated fabric after one year's exposure was 67%, and 93% after two years. The protection afforded by heavy-metal and mercury compounds was not entirely satisfactory, and of the purely organic compounds the phenolic type afforded the greatest resistance to micro-biological attack.

H. K.

Fire Retardant Coatings for Fabric Covered Aircraft. S. G. Weissberg, G. M. Kline, and H. L. Llansberry. *Ind. Eng. Chem.*, 41, 1742-1749 (Aug. 1949).

In the rapid evaluation of the relative performance of fire-retardant coatings on aircraft fabrics, a small centrifuge draws air through a small funnel-shaped entrance, the base of which accommodates the fabric under test. A Méker burner is so arranged in relation to the air stream that its flame lies on the coated surface. Fire-retardant coatings on cellulose nitrate dope are not sufficiently effective. Borax-boric acid mixture is effective as a fire retardant but has a deleterious effect on dope-fabric adhesion. Several fire-retardant coatings having good resistance to weathering are recommended for further experiment.

H. K.

Influence of Aftertreatments on the Wet Strength of Formaldehyde- and Chrome-hardened Casein Fibres. H. Nitschmann, L. Karasoy, and R. Signer. *Textil-Rund.*, 4, 182-186 (May 1949).

During experiments on the dyeing of casein fibres which had been hardened with formaldehyde and basic chromium salts an increase in the wet strength of the material was sometimes observed. This was traced to the influence of formaldehyde additions which were made to the dyebaths in order to diminish the removal by hydrolysis of the originally bound formaldehyde. Aftertreatments were carried out over a pH range using various buffers with and without an addition of formaldehyde. The final wet strength was found to depend on both the pH and the particular buffer used, but under all conditions addition of formaldehyde increased the strength. Under the most favourable condition of pH 5.8 with acetate buffer the wet strength increased 41%. Further experiments showed that the strengthening action increased with decrease in the buffer concentration, provided that the pH was maintained about 5.7, and that the maximum effect was attained with a formaldehyde concentration of 1-2%. The strengthening action was not observed at all with fibres which had been hardened with formaldehyde alone or with formaldehyde and aluminium salts.

F. A.

Theoretical and Practical Aspects of Drying. H. Netz. *Textil-Praxis*, 5, 11-15 (Jan. 1950).

The drying of textile materials by different methods is discussed mathematically with a view to giving equations for the influence of drying conditions on the final results.

B. K.

Behaviour of Cellulose Fibres on Drying. A. Schaeffer. *Textil-Praxis*, 5, 3-6 (Jan. 1950).

Dry cellulose fibres show damage on subsequent storing when treated at temperatures over 80°C. Wet, neutral cotton, however, can be dried at 100-120°C. without damage, but if alkali is present the temperature must not exceed 65°C. Repeated treatment in water followed by drying causes fibre damage. Additions of such products as Calgon, Trilon A and B, Igepon, or Gardinol to the last rinsing bath decrease the tendency of the fibre to degrade on drying, Gardinol being particularly effective if alkali is present.

B. K.

Imparting Dimensional Stability to Fabrics.

Munsingwear Inc. and F. Chatfield. *B.P.* 632,516.

The fabric is steamed and then fed between a pair of endless elastic belts running close to one another. The receiving ends of the belts are driven faster than the discharge ends, so that the belts contract and induce lengthways compressive shrinkage of the fabric.

C. O. C.

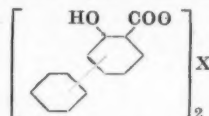
Lustering of Fabrics. Bleachers' Assocn. Ltd. and A. Melville. *B.P.* 632,655.

A woven fabric may be given a high degree of lustre as distinct from glaze, while retaining or even enhancing its weave characteristics, by applying a smoothing pressure or burnishing action between adjacent threads on one or both surfaces of the fabric. This can be done by supporting the fabric on a hard base and applying pressure to it by a comblike formation having resilient smooth-tipped blades relative to which the fabric is moved. The pressure is such that the blade tips press on to and below the normal surface of the fabric.

C. O. C.

Rotproofing of Cellulosic Textiles. F. J. Meyer and Dow Chemical Co. *U.S.P.* 2,480,084.

Impregnation with compounds of formula—



(X = Cu, Cd, Pb, or Zn), e.g. 1-8% by weight of cupric 3-phenylsalicylate, gives good protection even on exposure under adverse conditions for long periods.

C. O. C.

Fabric Resistant to Mustard Gas. I. Pöckel. *U.S.P.* 2,481,532.

Cloth impregnated with a resin made by heating a polyhydric alcohol, a drying-oil fatty acid, and a straight-chain dibasic acid containing > 5 C in the chain resists penetration by mustard gas or other vesicants.

C. O. C.

Presetting Nylon Hose. E. B. Bates. *B.P.* 631,448.

Nylon hose may be preboarded or plasticised by placing on metal forms carried by a continuous conveyor through a chamber in which a mixture of steam and hot air (at 230-260°C.) is maintained at atmospheric pressure. The forms are internally heated by electric elements, and during passage through the chamber pick-up contacts attached to the forms maintain a sliding engagement with electrical contacts for supply of current to the heating elements.

G. E. K.

Presetting Nylon Hose. Wolsey Ltd., P. Alexander, H. Burrows, and P. D. Smith. *B.P.* 631,456.

Presetting of nylon hose to obtain permanent loop formation and shape is carried out by knitting a pre-wetted yarn, and while the fabric is held under tension by the take-off mechanism directing air at 150-280°C. on to the knitted fabric in the vicinity of the needles.

G. E. K.

Adhesive Sheets or Tapes. Johnson & Johnson (Gt. Britain) Ltd. *B.P.* 592,218.

This patent has been amended, but the changes do not affect the abstract previously published (*J.S.D.C.*, 64, 89 (Feb. 1948)).

C. O. C.

Bonding Textile Yarns to Rubber. Courtaulds Ltd. and D. Entwistle. *B.P.* 632,109.

Improved bonding is obtained by vulcanising rubber in contact with yarns which have been impregnated with a compound of a metal, e.g. Zn, Cd, Hg, Cu, Ni, or Fe, the sulphide of which is insoluble in water.

J. W. B.

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Preparations of Dicarboxycellulose and its Esters.

A. Yashunskaya, N. Shorygina, and Z. Rogovin. *J. Appl. Chem. (U.S.S.R.)*, 22, 1037-1043 (Sept. 1949).

Following work on monocarboxycellulose and dialdehydocellulose and their esters (cf. *J.S.D.C.*, 66, 91 (Jan. 1950)), dicarboxycellulose preparations, formed from dialdehydocelluloses by oxidation with chlorous acid, have now been examined in a similar way. They are found to have a much lower solubility in dil. caustic alkali than the dialdehydocelluloses from which they are derived. The solubility of their nitric and acetic esters is considerably greater than that of the esters of the dialdehydocelluloses and approximates to the solubility of the esters of unoxidised cellulose. Esterification leads to a fall in carboxyl content, and it is suggested that lactones are formed by

the internal esterification of the erythric acid residues, while cross-linking, as postulated to explain the low solubilities of the esters of monocarboxycellulose and dialdehydocellulose, does not occur to an appreciable extent.

Permeability of Cellophane to Liquids. S. Madras, R. L. McIntosh, and S. G. Mason. *Canadian J. Res.*, B 27, 764-779 (Sept. 1949).

Permeabilities of several liquids have been measured at various degrees of swelling, which were obtained by suitable treatment of the Cellophane membrane. Degree of swelling as measured by thickness is retained when the swelling agent is removed by solvent exchange. For water and aqueous solutions the permeability coefficient K is about five times that of organic permeants. For homologous series of alcohols and ketones K decreases with increasing chain length. Attempts to calculate the effective pore radius and pore number were successful only for water and dilute NaOH solutions, where a radius of 1.5×10^{-7} cm. and a pore number of 10^{13} per sq. cm. were obtained. An independent method based on combined permeability and electrical conductance yielded a value of 3×10^{-7} cm. for the effective pore radius. The effect of viscosity of penetrant has been studied by measuring the permeability of water through the swollen membrane; evidence that flow is of a viscous type has been obtained. A rapid method of measuring water of imbibition based on a drying curve is described.

G. L.

PATENTS

Bleaching of Ground Wood Pulp. Du Pont.

B.P. 632,967.

An alkaline solution of a peroxide is used at pH 10.0-11.0 and 26-49°C.

C. O. C.

Paperlike Material from Calcium Alginate Rayon. J. R. Cairns, S. R. H. Edge, and Wiggins Teape & Co. (1919) Ltd.

B.P. 632,050.

Alginate rayon waste is pulped in a breaker or beater, and the diluted stock run on the paper machine. An alkali solution, e.g. sodium carbonate, is mixed with the alginate pulp stock in the head or breast box of the machine by introducing it into the mixing pump. It causes superficial swelling and softening of the fibre without weakening the interior, giving rise to improved bonding, and is drawn off again with the backwater drained and sucked from the wire. The backwater containing the alkali is returned to the head box as usual, and with a time of about 1 min. for the pulp to travel from the head box to the wire a concentration of 0.2-0.4% of sodium carbonate in the backwater is sufficient.

S. V. S.

Aqueous Dispersions of an Acid-reacting Synthetic Resin for Paper Sizing. Monsanto Chemical Co.

B.P. 632,024.

An aqueous dispersion of an acid-reacting synthetic resin made by a Diels-Alder reaction between an ethylene- β -dicarboxylic acid and a terpene (including diterpenes) is prepared by heating a mixture of the reaction product with an alkali and water under pressure at 140-200°C., preferably 160-170°C. The alkali employed is sufficient to neutralise 30-50% of the acid content of the resin, and the weight of water used is less than that of the reaction product. After heating for at least 30 min., the dispersion is allowed to cool to 60-90°C. before removal from the reaction vessel. It contains 50-80% by weight of reaction product, 4-14% of alkali, and 6-46% of water, and may be diluted to 1:15-16 by weight with water at 10-65°C. with agitation. The resin employed may be the reaction product of rosin and maleic anhydride, fumaric acid, citraconic acid, or itaconic acid or of maleic anhydride and dipentene, terpinene, or terpinolene.

S. V. S.

Paper. Papeteries Steinbach & Co. S.A. B.P. 632,218.

In manufacturing photographic paper, sizing is carried out by replacing the whole or most of the rosin size with at least one aldehydic condensation product of an aminotriazine, e.g. melamine resin. The melamine (0.25-5% of the weight of dry pulp) is added in hot aqueous solution to the beater in presence of formaldehyde. The appearance of uneven transparent spots in the course of developing and washing the paper after photographic use is thus avoided.

B.P. 632,267.

The method is modified for making heliographic paper by adding the melamine in aqueous solution either hot or weakly

acidified with HCl, in presence of formaldehyde, using 1-5% of the resin on the dry weight of pulp, preferably 3.0-3.5% for sulphite or soda pulp. The drying cylinders are kept substantially below 85°C., and the paper is afterwards brought to at least 85°C. for a short time before being reeled, to prime the polymerisation of the resin and to ensure that the paper temperature is at least 45°C. after reeling to allow polymerisation to be completed. S. V. S.

Salts of Phenol-Formaldehyde Condensates. C. J. Pedersen.

B.P. 629,467.

Stable salts of condensates formed by treating 2 mol. of formaldehyde with 1 mol. of phenol in aqueous solution are produced by stopping the reaction when addition products or partial condensates thereof have been formed, evaporating the water, and neutralising the phenol with an equivalent amount of base. They can be used as sizes for paper, impregnating agents, etc.

W. G. C.

Protective Coating on Carbon Paper. Kolok Manufacturing Co. Ltd. and E. S. Dumont.

B.P. 631,373.

Coating the dope surface of carbon paper with a material which melts at the temperature of application, the molten material in turn softening the dope and becoming adherent to it before the protective material solidifies, gives protection against atmospheric conditions and yields a cleaner-handling paper.

C. O. C.

Coated Sheet Material. Copeman Laboratories Co. and H. Y. Jennings.

B.P. 632,074.

A uniform film of rubber latex is applied to one side of a paper backing sheet by passing the sheet against a coating roller which dips into an aqueous dispersion of the latex and which rotates in a direction opposite to that of the sheet. The coating is partly heat-cured and the material rolled up to prevent further curing.

B.P. 632,141.

Latex-coated backing material is cured to a substantially non-tacky state and rolled up tightly to maintain cohesive properties, so that on unwinding there is no transfer of latex from one side to the other but the film as a whole may be easily separated from the backing sheet. The film is used as a protective coating with or without the backing material.

J. W. B.

Methylolmelamine Ether Resins modified with Orthoesters of Silicon. S. H. Rider and Monsanto Chemical Co. (XIII, p. 252.)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

PATENTS

Improving the Fastness to Rubbing of Dyed Suède Leather. D. T. Kirby, J. T. Chain, and Du Pont.

U.S.P. 2,481,933.

Wet fully tanned suède leather is treated with an aqueous dispersion of a water-insoluble synthetic polymer applied from an aqueous bath containing acid and/or direct dyes or from an aqueous bath containing a sulphonated-oil fat liquor. It is then further processed in the usual manner. Besides better fastness to rubbing, the fullness, weight, and firmness of the leather are increased.

C. O. C.

Coating Leather. United Chrometanners Ltd., H. Kacser, J. Utitz, and E. E. Utitz.

B.P. 631,451.

Leather is partly opened, e.g. by fluffing or wheeling, and then coated on one or both sides with a liquid dispersion of a thermoplastic resin in a plasticiser, if desired with addition of colouring material; it is then heated and pressed so as to fix the resin by gelation on and in the surface of the leather. The plasticiser must be non-volatile, a non-solvent at low temperatures but a solvent at higher temperatures. This gives a waterproof finish without impairing flexibility and improves the tensile strength and other physical qualities of the leather.

C. O. C.

Drying Hides, Skins, and Leather. E. A. Wagner.

U.S.P. 2,479,440.

The wet hide is placed on a magnetisable sheet, to which it is secured by a number of magnets placed along the edges of the hide and in its central portions. The magnets are of such strength and number that the hide can shrink 0-5% during drying.

C. O. C.

XIII—RUBBER; RESINS; PLASTICS

Synthesis of Vinyl Esters of Polyvinyl Alcohol. S. N. Ushakov and S. I. Kirillova. *J. Appl. Chem. (U.S.S.R.)*, **22**, 1094-1102 (Oct. 1949).

Polyvinyl alcohol was vinylated by treating in aqueous alkaline soln. with acetylene (12-18 atm., 100-115°C., 10-20 hr.). With higher temperatures or longer times a dehydration side-reaction occurred. Water- and spirit-soluble products, containing 3.5-6 vinyl radicals per 100 vinyl alcohol units, were obtained. A. E. S.

Relation of Physical Characteristics and Chemical Structure of Cellulose Derivatives. E. Ott. *Chem. and Ind.*, **53**, 915-922 (31st Dec. 1949).

The fundamental properties of organic high polymers suitable for plastics are explained by the application of normal chemical and physical principles, and apparent anomalies in the correlation of theoretical and experimental data are clarified. Using cellulose as a typical long-chain type of polymer, the nature of the intermolecular forces and rigidity of this chain molecule are summarised. Modification of the cellulose molecule by introduction of substituent groups, and the profound influence of the nature, size, and distribution of the substituent, as well as of the degree of substitution, on such properties as moisture sensitivity, softening-point, tensile strength, and solubility are discussed. An account of swelling and association-compound formation as preliminaries to solution, and theoretical considerations of the action of non-solvent plasticisers, are included. H. H. H.

PATENTS

Stabilising Polyethylene. Du Pont. *B.P.* 632,270.

Polyethylene is stabilised, without discoloration, against deterioration of mechanical and electrical properties by incorporating 0.001-5% of a compound of general formula $X-S-CH_2-CH_2-COOR$ ($R = \text{Alk or Ar of } > 3 \text{ C, } X = \text{hydrocarbon radical which may contain O and/or S, or S and N, e.g. dialkyl thio-}\beta\text{-dipropionate. E. C.}$

Films of Plasticised Vinylidene Chloride Copolymers soluble in Organic Solvents. British Cellophane Ltd. *B.P.* 632,114.

Use of a polyoxyalkylene ether of a ring dehydration product of a hexitol as the plasticiser results in sheets or coatings of exceptional flexibility, toughness, and durability. C. O. C.

Methylolmelamine Ether Resins modified with Orthoesters of Silicon. S. H. Rider and Monsanto Chemical Co. *U.S.P.* 2,482,508.

The resins obtained by heating 1 mol. of an alkyl ether of methylolmelamine with 0.2-3.0 mol. of an alkyl orthosilicate are soft, rubbery, and generally water-white. They have a very low viscosity coefficient, being extremely stiff and viscous at 150°C. yet still soft and rubbery at 30°C. They are compatible with alkyl resins, and can be partly cured without catalysts at 120-300°C. to yield hard, glossy, flexible films. The original and the partly cured resins are softened slightly by water and are soluble in aromatic hydrocarbons, but they recover their original hardness on drying. Heating at 100-200°C. in presence of an acid catalyst yields insoluble, infusible, water-inert products. They are used *inter alia* for impregnating paper, textiles, etc. C. O. C.

Ureide-Formaldehyde Condensates. Geigy Co. Ltd., H. Jones, and J. K. Aiken. *B.P.* 631,200.

Water-soluble condensates which can be used in moulding powders and for coating and adhesive purposes are obtained by treating trichloroethylendenediureide—



with formaldehyde. W. G. C.

Polymerisation of Glycol Dimethacrylates. Du Pont. *B.P.* 631,844.

The rate of polymerisation of these esters is considerably increased by adding < 1.5% of a combined polymerisation catalyst comprising 0.04-1% cobalt nitrate hexahydrate and 0.05-1% of a free-radical catalyst, e.g. *aa'*-azodiisobutyronitrile. W. G. C.

Distribution of Water-dispersible Material throughout Thermoplastic Polymers. J. A. Connell and Du Pont. *U.S.P.* 2,480,821.

Water-dispersible material, e.g. colouring material, is rapidly and cheaply distributed throughout a thermoplastic polymer by mixing an aqueous suspension of the

material with granular thermoplastic polymer, of particle size 0.1-2.0 mm., so that the total water content of the mixture is 0.15-0.9% on the weight of the polymer, and then masticating at not below the flow temperature of the polymer until the mixture is colloidal. C. O. C.

Resins from 3:5-Diamino-4-dihydrothiadiazine-1-dioxide for treating Textiles, Stoving Lacquers, etc. H. A. Walter and Monsanto Chemical Co. (III, p. 239.)

Vinyl Resin Stabilisers. U.S. Rubber Co. (III, p. 240.)

Solutions of Vinylidene Copolymers. Société Rhodiacéta. (VI, p. 246.)

XIV—ANALYSIS; TESTING; APPARATUS

Determination of Water by Karl Fischer Reagent. W. Seaman, W. H. McComas, and G. A. Allen. *Anal. Chem.*, **21**, 510-512 (April 1949).

The Karl Fischer method of estimating water is modified by replacing the single solution (containing sulphur dioxide, pyridine, methanol, and iodine) by two—one containing pyridine, sulphur dioxide, and methanol, in which the sample is dissolved or suspended; and the other containing iodine and methanol, which is used for titration. This method avoids decomposition of the reagent and eliminates the necessity for frequent restandardisation, without affecting the precision of the technique. A. S. F.

Phenyl- α -naphthylamine-azobenzene- p -sulphonic Acid as Adsorption Indicator. R. C. Mehrotra. *J. Indian Chem. Soc.*, **26**, 511-514 (Nov. 1949).

Phenyl- α -naphthylamine-azobenzene- p -sulphonic acid has been suggested as a new adsorption indicator for argentometric titrations, and differs from all the classical adsorption indicators in having both acidic and basic characteristics. This enables titrations of halide ions against silver ions as well as the reverse. The adsorbability by both positively and negatively charged silver halide bodies endows the indicator with a sharp colour change at the end-point, ready reversibility, and a wide range of applicability. H. H. H.

Quantitative Colorimetric Microdetermination of Methanol with Chromotropic Acid. R. N. Boos. *Anal. Chem.*, **20**, 964-965 (Oct. 1948).

The alcohol is oxidised with acidified permanganate (excess of which is destroyed with bisulphite) to formaldehyde, which gives with chromotropic (1:8-dihydroxynaphthalene-3:6-disulphonic) acid an intense violet-red colour permitting rapid quantitative colorimetric determination with accuracy within 2%. Most common aldehydes give no colour reaction; glyceraldehyde gives a yellow colour. The method is more convenient than the Zeisel method, and may be applied to determining methoxyl in methyl esters after hydrolysis. H. I. S.

Colorimetric Determination of Small Quantities of Formaldehyde by means of Chromotropic Acid. T. Kleinert and E. Srepl. *Mikrochem.*, **33**, 328-332 (1948); *Analyst*, **74**, 610 (Nov. 1949).

Formaldehyde yields a violet-red colour when warmed with chromotropic acid in 72% sulphuric acid. The reaction is selective for formaldehyde in the presence of many other aldehydes, e.g. acetaldehyde, propionaldehyde, *n*- and *iso*-butyraldehyde, *isovaleraldehyde*, cinnamal, crotonaldehyde, chloral hydrate, glyoxal, aromatic aldehydes, and furfural. The foreign aldehyde may be in considerable excess. The qualitative reaction described by Eegrove (*Z. anal. Chem.*, **110**, 22 (1937)) shows considerable variations in colour intensity, and various factors are investigated to render the method suitable for quantitative work. The resulting procedure is suitable for 0.002-0.05 mg. of formaldehyde, and the error varies from 2 to 5% when the Lange colorimeter is used. M. E. W.

Polarographic Determination of Ethylenediaminetetra-acetic Acid. W. Furness, P. Crawshaw, and W. C. Davies. *Analyst*, **74**, 629-635 (Dec. 1949).

The polarographic characteristics of the ferric and cupric complexes of ethylenediaminetetra-acetic acid are reported. The latter is found to be particularly suited for the purpose of evaluation of commercial samples of the sodium salt of the acid (Trilon B), and in the quantitative method devised, the complex, formed in the presence of an excess of cupric ion, is determined polarographically

after precipitating the excess of copper with magnesium oxide. The polarographic results are compared with those obtained by an independent photometric procedure and by the Kjeldahl method. H. H. H.

Estimation of Non-ionic Detergents. J. Oliver and C. Preston. *Nature*, 164, 242-243 (6th Aug. 1949).

Non-ionic detergents, etc. of the polyethylene oxide condensate type may be estimated gravimetrically by precipitation with 10% phosphomolybdic acid in the presence of 10% barium chloride solution and dilute hydrochloric acid, the method having good reproducibility. Sulphates interfere and if present are first removed by precipitation with barium chloride solution. W. K. R.

X-Ray Diffraction Patterns for the Identification of Surface-active Agents. T. F. Boyd, J. M. MacQueen, and I. Stacy. *Anal. Chem.*, 21, 731-732 (June 1949).

The X-ray diffraction pattern of a surface-active agent may be used as a means of rapid identification. Diffraction data are presented for 19 crystalline compounds of different chemical types separated from commercial samples by alcohol extraction. W. K. R.

Polarographic Determination of Benzene in presence of its Homologues. A. S. Landry. *Anal. Chem.*, 21, 674-677 (June 1949).

Benzene is determined in the presence of toluene and xylene by nitration, with subsequent selective oxidation of the toluene and xylene nitration products and isolation of the dinitrobenzene by differential solubility extraction. The dinitrobenzene is then polarised in the presence of a suitable base, and the amount present in the sample estimated from a prepared standard curve. W. K. R.

Rapid Colorimetric Estimation of Phenol. R. W. Martin. *Anal. Chem.*, 21, 1419-1420 (Nov. 1949).

The reddish colour produced by the condensation of 4-aminoantipyrin with phenols in the presence of alkaline oxidising agents, e.g. potassium ferricyanide, has been made the basis for the rapid absorptiometric estimation of phenol, in the range 0.2-2.0 p.p.m., in either water or concentrated salt brines, with an accuracy of $\pm 10\%$. The reaction is a general one for phenols, since cresols, xylenols, naphthols, etc. each produce a characteristic colour, and these substances, when present, will interfere with the phenol determination. H. H. H.

Determination of Phenol and *m*-Cresol in Complex Biochemical Mixtures—Counter-current Distribution Method. B. Warshowsky, E. J. Schantz, and M. W. Rice. *Anal. Chem.*, 20, 951-954 (Oct. 1948).

The phenolic compounds are removed from biochemical or industrial mixtures by distillation at pH 8.0-8.5, and the sum of phenol and *m*-cresol is determined colorimetrically in an aliquot by the diazotised sulphanilic acid method. Phenol is then separated from cresols in an aliquot by extraction from aqueous 0.1M. potassium hydrogen phthalate pH 5.0 buffer with carbon tetrachloride, using a Craig 24-plate counter-current distribution machine. From the phenol contents of a selected series of extraction tubes, the concentration in the original sample is calculated. *m*-Cresol is determined by difference. Some volatile phenolic compounds (e.g. 2-hydroxy-1:4-dimethylphenol) interfere. H. I. S.

Colorimetric Analysis of *m*- and *p*-Cresol in their Mixtures. S. A. Savitt, A. M. Goldberg, and D. F. Othmer. *Anal. Chem.*, 21, 516-518 (April 1949).

A simple and accurate method of estimating the concentrations of *m*- and *p*-cresol in their mixtures depends on the difference in absorption characteristics of their nitroso-phenol complexes. A. S. F.

Fluorometric Determination of Malic Acid and 2-Naphthol. E. Leininger and S. Katz. *Anal. Chem.*, 21, 1375-1377 (Nov. 1949).

Based upon the reaction of malic acid and β -naphthol in sulphuric acid solution, separate methods for the determination of each of these two compounds are described which depend on the fluorescence intensity readings of a fluorescence meter. The effect of variations in the operating conditions for each method is discussed. The method for β -naphthol is applicable to determinations of 1-14 $\mu\text{g.}$, and the presence of α -naphthol in a 1:1 ratio with β -naphthol causes an error of $\sim 5\%$. H. H. H.

Determination of Carboxy Group in Aromatic Acids. M. H. Hubacher. *Anal. Chem.*, 21, 945-947 (Aug. 1949).

The carboxyl group attached to an aromatic nucleus may be estimated by measuring the amount of carbon dioxide liberated when the acid is heated with quinoline in the presence of a catalyst. An apparatus is described in which the estimation is carried out on samples of 0.002-0.02 mole of acid. A comparative study of inorganic catalysts showed that basic cupric carbonate was the most efficient in most cases, and where this failed silver carbonate was suitable. The accuracy and precision of the method are only fair, but it is of value in cases where determination by titration is not possible. W. K. R.

Report on Non-volatile, Unsulphonated, Amine Intermediates in Coal-tar Colours—Determination of 3-Nitro-*p*-toluidine [*o*-Nitro-*p*-aminotoluene] and *p*-Nitroaniline. L. S. Harrow. *J. Assocn. Offic. Agr. Chemists*, 32, 624-626 (1949); *Chem. Abs.*, 43, 9454 (25th Nov. 1949).

o-Nitro-*p*-aminotoluene in D&C Red No. 35 and 38 is estimated by extracting it from the dye with petroleum benzene and titrating with TiCl_3 . Results are given of a collaborative study of the determination of *p*-nitroaniline in D&C Black No. 1 (*Chem. Abs.*, 43, 7231 (1949)). C. O. C.

Report on Unsulphonated Phenolic Intermediates in Coal-tar Colours—Determination of 2-Naphthol in D&C Red No. 35. H. Holtzman and H. Graham. *J. Assocn. Offic. Agr. Chemists*, 32, 617-621 (1949); *Chem. Abs.*, 43, 9453 (25th Nov. 1949).

The dye is extracted with hot acid, the extract coupled with diazotised sulphanilic acid, and the resulting dye titrated with TiCl_3 . The conditions for extraction need further study. C. O. C.

Automatic Paper Chromatography. R. H. Müller and D. L. Clegg. *Anal. Chem.*, 21, 192 (Jan. 1949).

Rapid and complete separation of microgram quantities of dye mixtures has been obtained by the following technique—Filter-paper is embossed with paraffin to form restricted rectangular channels, in one of which a spot of the mixture is deposited. A fine spot of light is focussed on the paper at one side of the sample, and at the other side a suitable eluting solvent is added from a microcapillary. As each component passes the light spot the transmittance and reflectance of the paper are modified, and these changes are followed by focussing the emergent light on a photomultiplier and measuring the current produced on a recording potentiometer. W. K. R.

Report on Spectrophotometric Analysis of Coal-tar Colours—D&C Green No. 6. R. N. Sclar. *J. Assocn. Offic. Agr. Chemists*, 32, 635-640 (1949); *Chem. Abs.*, 43, 9452 (25th Nov. 1949).

Details are given of the determination of the dye in chloroform solution, such solutions being stable for three days if stored in the dark. C. O. C.

Report on Subsidiary Dyes in Coal-tar Colours—Spectrophotometric Determination of D&C Orange No. 4 in Samples of FD&C Orange No. 1. M. Dolinsky. *J. Assocn. Offic. Agr. Chemists*, 32, 640-644 (1949); *Chem. Abs.*, 43, 9452 (25th Nov. 1949).

A rapid spectrophotometric method for determining D&C Orange No. 4 in FD&C Orange No. 1 is given, being applicable to 1.0-9.1% mixtures of No. 4 with 99.0-90.9% of pure No. 1 with an average error of 11.1%. C. O. C.

Report on Acetates, Carbonates, Halides, and Sulphates in Certified Coal-tar Colours—Determination of Sodium Acetate in FD&C Blue No. 1. J. Schifferli and A. T. Schramm. *J. Assocn. Offic. Agr. Chemists*, 32, 614-617 (1949); *Chem. Abs.*, 43, 9452 (25th Nov. 1949).

The Sclar and Clark method (*Chem. Abs.*, 38, 6098 (1944)) has been modified. In 7 determinations of added sodium acetate an average difference of -0.0007% was obtained between the found and calculated values. C. O. C.

Report on Spectrophotometric Testing of Coal-tar Colours—Identification of Fluorescein Colours. R. N. Sclar. *J. Assocn. Offic. Agr. Chemists*, 32, 626-635 (1949); *Chem. Abs.*, 43, 9453 (25th Nov. 1949). Data on the wavelength of maximum absorption for

dilute ammoniacal solutions of the 15 certifiable fluorescein dyes are given. They enable identification of most of these dyes where only one dye of this type is present. In other cases, qualitative analysis for halogens or nitrogen will be needed to complete the identification. D&C Oranges No. 5 and 15 and D&C Reds No. 21 and 29 cannot be distinguished by this method; they can be identified from the spectrophotometric curves of their solutions in 50% alcohol at several pH values. Data on the position and magnitude of the absorption peaks of D&C Yellow No. 7, D&C Oranges No. 5 and 14, and D&C Reds No. 21 and 29 are given. C. O. C.

Report on Determination of Lead in Coal-tar Colours. N. Ettelstein. *J. Assocn. Offic. Agr. Chemists*, **32**, 622-623 (1949); *Chem. Abs.*, **43**, 9451 (25th Nov. 1949).

Collaborative study has shown the dithizone method to be satisfactory. C. O. C.

Report on Determination of Pure Dye in Lakes and Pigments. K. A. Freeman. *J. Assocn. Offic. Agr. Chemists*, **32**, 644-647 (1949); *Chem. Abs.*, **43**, 9452 (25th Nov. 1949).

Previous collaborative results on a method for determining pure dye (*Chem. Abs.*, **43**, 7231 (1949)) showed much variation, possibly due to lack of uniformity in standardising the TiCl_3 solution. Examination of 2 samples each of D&C Red No. 34 Ca Lake, D&C Red No. 14 Na Lake, and Ext. D&C Red No. 2 Ba Lake, made by 8 analysts using a uniform standardisation method for the TiCl_3 , gave good results both among the different workers and compared with the calculated values. C. O. C.

Report on Determination of Lead in Aluminium and Barium Lakes of Coal-tar Colours. L. S. Harrow. *J. Assocn. Offic. Agr. Chemists*, **32**, 621, 622 (1949); *Chem. Abs.*, **43**, 9452 (25th Nov. 1949).

Results of a collaborative study of methods for determining lead in aluminium and barium lakes of coal-tar dyes are given. C. O. C.

Evaluation of Red Lead Pigments—II. A. Jogarao. *J. Sci. Ind. Research (India)*, **7B**, (9), 138-144 (1948).

Detecting Arachis Oil in Olive Oil. J. Spiteri. *Chim. anal.*, **31**, 196-199 (1949).

Determination of Pyrogallol in Hair Dyes. C. R. Joiner. *J. Assocn. Offic. Agr. Chemists*, **32**, 592-598 (1949); *Chem. Abs.*, **43**, 9368 (25th Nov. 1949).

The efficiency of the A.O.A.C. method, modified to reduce the blank and eliminate the effect of alumina cream on development of the colour, has been confirmed by six independent analysts working on three different samples. C. O. C.

Application of Physical Methods to Analysis of Inorganic Pigments. R. Schmidt. *Vefskroniek*, **22**, 140-143 (1949); *Chem. Abs.*, **43**, 8697 (10th Nov. 1949).

Use of potentiometric, polarographic, and spectrographic methods not hitherto used for analysis of inorganic pigments simplifies and improves the latter in comparison with the methods usually used. Examples described are determination of the Zn content of lithopone by measurement of the redox potential of a $\text{K}_3\text{Fe}(\text{CN})_6$ - $\text{K}_4\text{Fe}(\text{CN})_6$ system and the method of Delahaye, polarographic determination of Cr^{3+} and Cr^{4+} in Berlin Blue, and determination of Pb, Zn, Ba, Ca, and Si in a mixed pigment by emission spectrography. C. O. C.

Use of Dyes to determine the Isoelectric Point of Gelatin. H. O. Dickinson. *Nature*, **163**, 485-486 (26th March 1949).

A quick and convenient method for determining the isoelectric point of gelatin is described which depends on the effect of small concentrations of gelatin on the absorption characteristics of aqueous solutions of certain carbo-cyanine dyes. Good agreement is obtained between this method and the spirit precipitation method. A. S. F.

Chemical Microscopy in Dyeing and Finishing. G. L. Royer. *Anal. Chem.*, **21**, 442-447 (April 1949).

The application of microscopical techniques to problems in dyeing behaviour, the identification of textile fibres, the penetration of dyes into fibres, and to finishing is described. The paper is illustrated by colour photomicrographs. A. S. F.

Photographic Aids to the Textile Industry. M. Siegel. *Amer. Dyestuff Rep.*, **38**, P 268-P 273 (21st March 1949).

The applications to the textile industry of high-speed still and motion picture photography, electron photomicrography, and photography using ultra-violet, infra-red, and X-ray techniques are briefly reviewed. H. K.

Quantitative Analysis of Fibre Mixtures. A. Agster. *Textil-Praxis*, **4**, 504-508 (Oct. 1949).

Practical details are given for the quantitative analysis of a large number of fibre mixtures. F. A.

Determination of Cellulose by Acid-Dichromate Oxidation. L. Segal, R. C. Tripp, V. W. Tripp, and C. M. Conrad. *Anal. Chem.*, **21**, 712-718 (June 1949).

It has been found that the volumetric acid-dichromate method for the determination of cellulose accounts for only about 97% of the theoretical weight of oven-dried, purified cotton cellulose. It is shown that the deficiency is due to —(1) the formation and escape of carbon monoxide equivalent to 1-66% cellulose during the oxidation, (2) the presence of 0-5-0-6% moisture which is not removed by conventional drying in an air oven at 103-110°C., and (3) residues of 0-10-0-15% ash which are not removed by the normal purification techniques. No acetic acid is produced during the oxidation of the cellulose. By the use of an experimental milliequivalent weight of 6-861 mg. in place of the theoretical value of 6-750 mg., derived from the observed relative amounts of carbon monoxide and carbon dioxide formed during the oxidation, accurate cellulose analysis may be accomplished. W. K. R.

Determination of Cellulose in Cotton and Cordage Fibre. L. E. Hessler and G. V. Merola. *Anal. Chem.*, **21**, 695-698 (June 1949).

The effect of alcoholic amines in ethylene glycol has been studied as a means of determining cellulose in cotton and cordage fibres. Results obtained by refluxing with a mixture of 1 part of monoethanolamine and 3 parts of ethylene glycol compared favourably with other methods of determining cellulose in cotton fibre. The action of this reagent on hard and soft fibres shows promise as a method for determining cellulose, but in some cases the fibre may require some bleaching. Comparisons of the action of various cellulose reagents on cordage fibres are given. W. K. R.

Examination of Textile Fibres by the "Dispersion Staining" Method. P. C. Douglass and G. C. Crossmon. *Text. Research J.*, **19**, 644-646 (Oct. 1949).

A dispersion staining technique for determining the structure and identity of textile fibres is described. A sample of fibre is mounted in a liquid of high dispersion having a refractive index near that of the fibre. The fibre is identified by the resulting coloration, using an ordinary compound microscope arranged for dark-ground illumination. Fibres which are classed as striated or microscopically structureless are distinguished by this technique without requiring any special preparation of the sample. P. C.

Determination of the Wax Content of Raw Cotton Fibre. C. Lesslie, L. Hagan, and J. D. Guthrie. *Anal. Chem.*, **21**, 190-191 (Jan. 1949).

Values for the wax content of raw cotton obtained by treatment with hydrochloric acid prior to extraction (cf. Fargher and Higginbotham, *J. Textile Inst.*, **18**, 283-287 (1927)) are considered to be invalid, as it is shown that the substances responsible for the increased wax values following such treatment are water-soluble but are converted by the acid into a material that behaves like wax on extraction. W. K. R.

Moisture Determination in Textiles by Electrical Meters—III. J. C. Whitwell and R. K. Toner. *Text. Research J.*, **19**, 755-758 (Nov. 1949).

Tests are described made on the Moisture Register, an electrical meter designed to measure the moisture content of wool textile materials. When used on wool it gives results accurate to $\pm 0.35\%$ moisture, but is less precise when applied to cotton sliver and acetate staple. As with other meters, it is sensitive to temperature changes and the presence of impurities in the material under test. P. C.

Spectrographic Determination of Chlorine in Textiles. W. W. A. Johnson and D. P. Norman. *Amer. Dyestuff Rep.*, 38, 361-363 (2nd May 1949).

Small amounts of chlorine (5 μ g.) in textiles may be determined spectrographically in the form of aluminium chloride; the emission spectrum of which at 2100 Å. is readily observed in vacuum. One sq. in. of fabric is shredded, mixed with 0.1 g. of chlorine-free aluminium powder, placed in a small brass cup attached to the lower tungsten electrode in a discharge tube, and subjected to a discharge (> 1000 v., > 1 amp.) under vacuum. Two methods are described for calibrating the observed brightness of the aluminium chloride band in terms of mg. of chlorine per sq. in. of fabric. In view of the small sample required, the method is suitable for determining local variations in the chlorine content of fabrics, etc.

W. K. R.

Methods for the Investigation of Formaldehyde-treated Fibres. A. Schaeffer. *Textil-Praxis*, 4, 287-289 (June 1949).

Practical details are given of the carbazole test for the detection of formaldehyde in fibres. The formaldehyde may arise from various finishes, such as Kaurit WF110, Kaurit KF, or thiourea- or melamine-formaldehyde condensates. The type of finish can be determined from further tests for nitrogen, urea, sulphur, and melamine, and full details are also given for these tests.

F. A.

Application to Viscose Rayon of the Differential Dyeing Test for Cotton Maturity. W. Arnfield and J. Boulton. *Text. Research J.*, 19, 212-214 (April 1949).

The differential dyeing test for cotton maturity devised by Goldthwait, Smith, and Barnett (*Textile World*, 97, 105 (July 1947)) has been applied to viscose rayon staple. Staple of mixed deniers is dyed with a mixture of Diphenyl Fast Red 7BL (C.I. 278) and Chlorantine Fast Green BL, for 45 min. at the boil, when fine-denier fibres appear green and coarse fibres (50 denier) are dyed red. An alternative mixture of dyes is suggested. When applied to viscose (filament denier 5-6) and cuprammonium (filament denier 1-33) rayons, the former is dyed red and the latter green. Modified viscoses produced by steaming normal viscose under pressure give rise to different hues according to the degree of treatment.

P. C.

Index of the Water-repellency of Textiles from the Surface Tension of Aqueous Solutions. H. A. Schuyten, J. W. Weaver, and J. D. Reid. *Amer. Dyestuff Rep.*, 38, 364-366, 368 (2nd May 1949).

The "rolling drop" method of evaluating the water repellency of fabrics is described. A range of solutions differing in surface tension by 1 dyne/cm. are prepared by dissolving wetting agents along with electrolyte in water. Solutions of decreasing surface tension (0.5 ml.) are successively applied dropwise and allowed to roll down the surface of the pattern (5 × 1 in.), which is inclined at 45°; wetting is indicated by the closing of an electric circuit. The highest surface tension required to cause wetting is taken as an index of water repellency. The method is rapid, gives reproducible results, and is capable of detecting small differences in water repellency not shown by other methods. Results obtained by a comparison of this and four other water repellency tests on eleven fabrics are quoted.

W. K. R.

Effective Laboratory Evaluation of Textile Detergency. E. A. Leonard and A. R. Winch. *Rayon Synthetic Text.*, 30, 79-81 (Jan.) and 93-95 (Feb. 1949).

The performance of detergents in continuous-flow systems has been determined quantitatively on a laboratory scale. Four stainless steel beakers were used as bowls for the scouring of wool yarn, produced from carded and spun raw wool in the grease. 5-g. hanks were agitated for 30-sec. periods in the liquors, which were maintained at a constant temperature throughout the test. The residual grease in the dried hanks was determined by a rapid extraction in carbon tetrachloride. Analysis of the results obtained provides a method of evaluating not only the detergents but factors such as temperature, nip pressure, etc. With slight modification the apparatus can be used to study scouring on the countercurrent principle.

H. K.

Flax Wet Spinning Frame as a Source of Catalytic Damage in Bleaching. W. Honneyman. *Chem. and Ind.*, 39, 663-667 (24th Sept. 1949).

Damage resembling cutting occurs sometimes in linen fabrics due to the catalytic oxidation of cellulose by hypochlorites and peroxides during bleaching in the presence of copper compounds. Since copper, particularly in the form of sulphide, is difficult to remove other than by the use of sodium cyanide, it is desirable to prevent its access to the yarn. Experiments have shown that an electric potential is set up between various parts of the trough of the wet-spinning frame by copper and brass acting as the poles of an electric cell. A self-generated electric current passes from one frame to another through the steam and water connecting pipes, and corrosion of the copper takes place, probably proportionally to the conductivity of the water in the trough and the length of time the water has been in the trough since the last cleaning out. The use of copper and brass together is unsatisfactory on account of the corrosion of the copper and consequent contamination of the yarn. Stainless steel is recommended for use throughout, with brass as a second choice. Whilst slight corrosion of the brass may occur, the effect is small compared with that produced when copper and brass are used together. The spinning frames should be electrically insulated from each other.

H. K.

Levelness of Acid Wool Dyes—Application of Laboratory Tests to Production. E. A. Leonard, G. A. Lathrop, and E. P. Mersereau. *Text. Research J.*, 19, 638-643 (Oct. 1949).

A quantitative laboratory method of evaluating the level-dyeing properties of acid wool dyes is described. It consists in dyeing a skein of yarn for half the normal dyeing period and then making additions of white yarn and fresh dye liquor before completing the second half of the dyeing time. The reflectances of the two dyed yarns and of undyed yarn are determined, and a "levelness index" is calculated. For dyes having ideal levelling properties the value of the index is 1, whilst for absence of levelling it is 0-333. Comparison of the levelness indices with dyeing experience shows that dyes having indices lower than 0.6 are not suitable for skein dyeing, whereas those having indices above 0.7 are suitable, other dyeing conditions being satisfactory. The effect of salt concentration on levelness index is generally in accord with dyeing experience. A mill test is described in which the levelness of 500 lb. Hussong stock-dyed batches was evaluated.

P. C.

Trade Practice Rules and Test Procedures for Textiles. H. Miller. *Amer. Dyestuff Rep.*, 38, P 274-P 275, P 280 (21st March 1949).

The Federal Trades Commission's requirements in the labelling of wool, silk, linen, and rayon textiles indicating the percentage of each fibre present are outlined, and the desirability of having standard test methods and procedures for determining whether these goods conform to the specified requirements is emphasised.

H. K.

Critical Observations on Fastness Testing. A. Schaeffer. *Textil-Praxis*, 4, 233-237 (May 1949).

The work of the British, German, Swiss, and American Committees on fastness testing is reviewed and the lack of co-ordination regretted. Various criticisms are then made of existing tests. Large pieces of apparatus, such as the wash wheel, should be avoided. The tests should not only be as simple as possible but their description should also be simple, this desideratum being illustrated by examples. The weight of material should be specified as well as the liquor ratio. It should be possible to carry out the tests on yarn as well as fabric. Tests on synthetic fibres should be included, and also tests on fastness to the new resin finishes.

F. A.

Assessment of Fastness Properties of Dyed Textiles. Anon. *Teintex*, 14, 519-525 (Dec. 1949).

Following the publication of a standard method for the assessment of fastness to light (*ibid.*, 14, 312 (July 1949)), the Fastness Committee of the Institut Textile de France has published standard methods for assessing fastness to washing, milling, rubbing, carbonising, water, sea-water, hot-pressing, and wet-pressing of dyed textiles. Tests are to be carried out at the auxiliary type depths proposed for the preparation of dyeings for fastness-to-light estimations, and bleeding is to be assessed against a grey scale which has still to be published.

B. K.

Fastness Properties of Coloured Textiles. C. Zuber. *Teintex*, 15, 9-29 (Jan. 1950); *Textil-Rund.*, 5, 15-19 and 41-49 (Jan. and Feb. 1950).

A discussion on standard methods for the evaluation and assessment of fastness properties of coloured textiles put forward by the S.D.C., D.E.K., I.C.I., and A.A.T.C.C. Fastness to light, bleaching, washing, milling, and rubbing is discussed, and the apparatus suggested for their evaluation is described together with the use of grey scales for assessing the results. B. K.

Assessment of Fastness to Light of Dyeings and Prints. Institut Textile de France. *Teintex*, 14, 312-313 (July 1949).

For assessment of fastness to light eight standards have been proposed, which are stated to correspond closely with B.S. 1006; Part 2—

- (1) 0.6% Brilliant Milling Blue BN
- (2) 0.8% Brilliant Acid Blue JR
- (3) 0.9% Brilliant Acid Blue 6B
- (4) 1.75% Supracide Blue J
- (5) 1.5% Acid Alizarine Sapphire R
- (6) 2.5% Acid Alizarine Sapphire 2JL
- (7) 1% Indigo 4B powder
- (8) 3% Indigosol AGG (DH)

The test samples are to be exposed under glass in a case inclined at an angle of 45°, facing south, in a pure atmosphere. Twelve standard-depth dyeings on wool gaberdine and bright viscose are employed in preparing patterns for exposure. B. K.

Factors influencing Crockfastness of Package-dyed Naphthol Cotton Yarns. American Association of Textile Chemists and Colorists, South Central Section. *Amer. Dyestuff Rep.*, 38, P 786-P 788 (31st Oct. 1949).

The effects of yarn preparation, manner of naphtholation, manner of developing, soaping, and softening on the rubbing fastness of package-dyed naphthol yarns have been measured experimentally. A. S. F.

Artificial Daylight—A New Colour Matching Unit—I. W. Harrison. *J. Textile Inst.*, 40, P1025-P1030 (Nov. 1949).

The required characteristics of an illuminant suitable for the matching and grading of coloured samples are stated. A lamp with improved characteristics is the new Siemens daylight matching unit, which utilises two fluorescent lamps in conjunction with two incandescent single-coil gas-filled filament lamps. From the window of the apparatus, which is approx. 24 in. x 18 in., an illumination of 45 lumens per sq. ft. is obtained, on a working plane 3 ft. below the window. H. K.

Artificial Daylight in the Textile Industry—II. H. Hewitt. *J. Textile Inst.*, 40, P1031-P1035 (Nov. 1949).

The chief requirements for accurate colour matching are—(a) a colour-matching unit, such as the Siemens unit, which apart from supplying light of good colour and constant spectral energy distribution, should provide a large area of low brightness; (b) normal colour vision in the person responsible for colour matching; (c) working conditions which favour accurate colour vision, since accurate colour matching is rendered difficult by mental strain and eye fatigue; (d) the surroundings adjacent to the matching position should be chosen to eliminate discrepancies due to "simultaneous contrast"; i.e. if two contrasting hues are in close proximity, each appears to

become tinged with the complementary hue of the other (neutral gray is preferred as a background against which samples are matched); (e) arrangement of the colours to minimise "successive contrast", i.e. the modification of a colour when it is viewed immediately after a contrasting colour (it is customary to commence matching greys and then proceed to the brighter colours if a wide range has to be matched); (f) a source which enables the person matching samples to judge by both artificial light, i.e. incandescent tungsten-filament lamps, and near natural daylight, i.e. a combination of incandescent and fluorescent lamps. H. K.

Assessment of Shrinkage in Washing. Schweizerischer Verband für die Materialprüfungen der Technik, Kommission 25, Richtlinienblatt C 3004. *Textil-Rund.*, 4, 462-463 (Dec. 1949).

An apparatus is described for testing the shrinkage of yarns in washing. A hank of standard weight and length is suspended between two rollers set at standard pressure and rotates under specified conditions with one half of its length immersed throughout the test in the scouring liquor. After treatment, the hank is air-dried and conditioned and then compared for length with an untreated hank. The scouring liquor consists of a 0.5% soap solution; treatment is carried out for 15 min. at 40°C., and is followed by rinsing under specified conditions. B. K.

Action of Light on Textile Materials. D. K. Appleby. *Amer. Dyestuff Rep.*, 38, 149-156, 189-192 (21st Feb. 1949).

This literature survey of work since 1920, containing 232 references, deals with the effect of light on the physical properties of various textiles. The parts played by the light source and exposure conditions are first pointed out. The changes produced by irradiation in each important natural, semi-synthetic, and synthetic fibre are then surveyed. A. S. F.

Hypochlorite Oxidation of Cotton Cellulose in presence of Mixtures of Certain Vat Dyes—L. G. M. Nabar and J. A. Rathod. *J. Sci. Ind. Research (India)*, 8B, (5), 81-86 (1949); *Chem. Abs.*, 43, 8671 (10th Nov. 1949).

Reduced Ciba Blue 2B does not accelerate oxidation of cellulose by dil. NaOCl; Cibanone Orange R under similar conditions causes considerable acceleration of oxidation. In presence of reduced Ciba Blue 2B, reduced Cibanone Orange R loses its identity, with the result that there is no acceleration of oxidation by NaOCl. C. O. C.

PATENTS

Testing Crease Resistance. L. Boor, L. A. Fluck, and American Cyanamid Co. *U.S.P.* 2,479,303.

The sample is folded upon itself and over a supporting edge. Standard pressure is then applied to the fold for a standard time, and then the angle formed by the sample on release of the pressure is measured without removing the sample from the supporting edge. C. O. C.

Testing Crease Resistance. R. D. De Waard, C. R. Stock, and American Cyanamid Co. *U.S.P.* 2,482,470.

The sample is folded upon itself, given standard pressure for a standard time, and then hung over a support, and the angle formed by the folded sheet is measured. C. O. C.

Emulsifying Power of Detergents. J. P. Sisley, Reutenauer, and Sicard. (III, p. 238.)

Replica Studies of Dyed Nylon. F. A. Hamm and J. J. Comer. (VIII, p. 247.)

Laboratory Evaluation of Sizing Materials for Nylon Hosiery Yarns. H. B. Goldstein. (X, p. 249.)

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